# The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 69 Number 7

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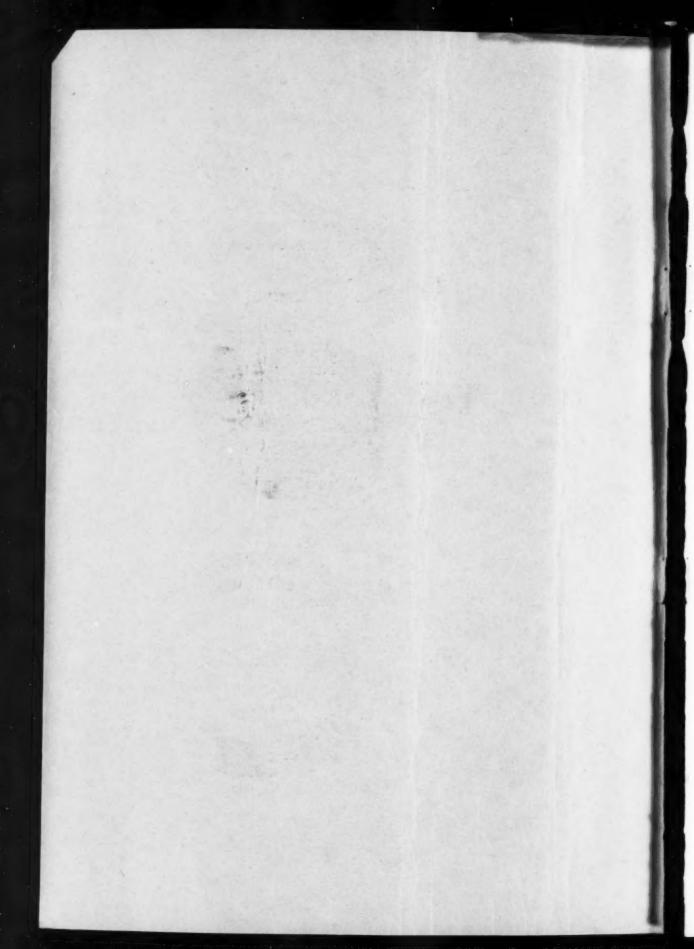
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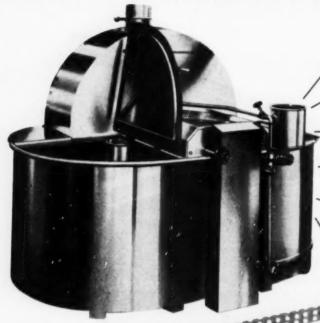
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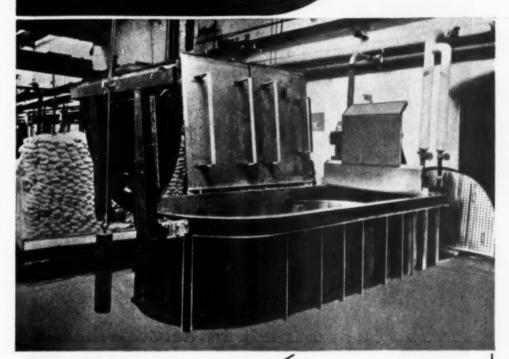
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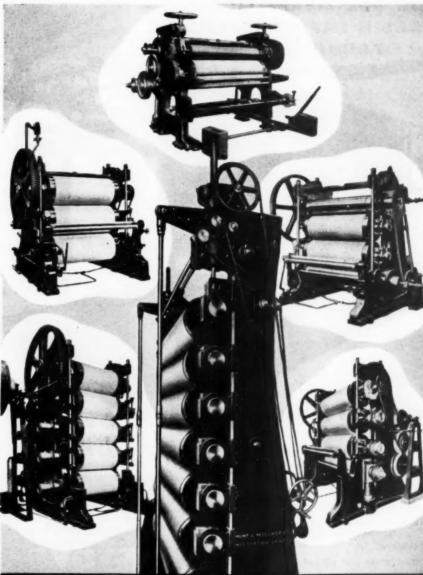
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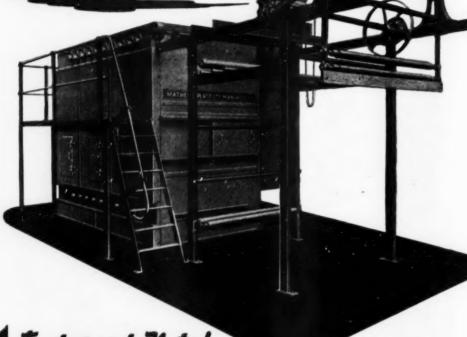




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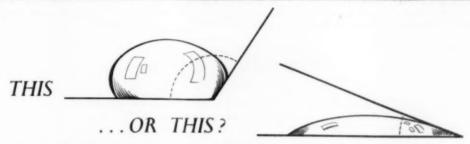
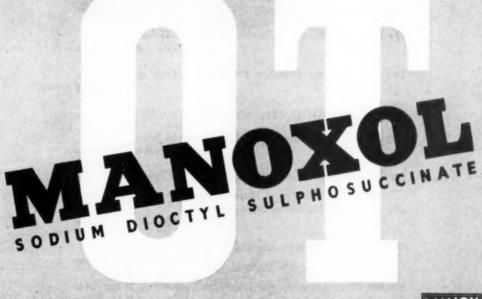


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#### Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the Journal-

#### LECTURES

- The Textile Trade and the Newer Fibres
- F. H. Clayton
- Ancient and Modern Textile Production in London and the Home Counties Sir Ernest Goodale
- Radioactive Isotopes in Printing W. Meitner and H. B. Hampson
- Studies in the Printing of Terylene Polyester Fibre A. G. H. Michie
- Fugitive Tinting with particular reference to Celafibre H. C. Olpin and A. J. Wesson
- Fuel Efficiency in the Dyeing and Finishing Trades W. Short
- The Uses of Microscopy in Textile Dyeing and Finishing
  G. G. Taylor and J. C. Brown
- The Structure and Chemical Properties of Dyes in relation to their Use

  T. Vickerstaff

#### COMMUNICATIONS

- Polymerisation of a Series of Vinyl Monomers in Wool

  N. K. Boardman and M. Lipson
- Studies in the Fundamental Processes of Textile Printing.
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- Observations on Some Commercial Colour Matching Lamps

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- Further Observations on the Effects of Evaporating Water from Cotton Cellulose

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#### Proceedings of the Society

#### THE THIRD GEORGE DOUGLAS LECTURE

## The Search for New Dyes in relation to Modern Developments in the Textile Field

T. HOLBRO

Meeting held in the Chemistry Department, University of Leeds, on 27th March 1953, Mr. H. H. Bowen (President of the Society) in the chair

I feel it a signal honour to be invited to deliver the third George Douglas Lecture, and it is a particular pleasure to be able to address you here in Leeds, a centre of research to which dye chemistry owes so much. I fully realise that the reason a dye chemist has been chosen to give this address is that problems arising in the field of textile dyeing and printing, both now and in the future, can be solved only by the combined efforts of all those connected with this field. In particular, I should like to emphasise the importance of exchanging ideas between colourists, specialists in application research, textile chemists and engineers. specialists in auxiliary products and finishing agents, and dye chemists. It is with this thought in mind that I have ventured to choose "co-operation" as the keynote for this lecture, which is well in keeping with the example set us by George Douglas.

Before attempting to discuss what I consider to be the most important problems in the field of dyes for the textile industry, I should like to make a brief survey of the present position of dye chemistry. In doing so, I may remind you of the vivid picture Dr. H. Levinstein painted in the first George Douglas lecture of the pioneer work and advances made in this field <sup>1</sup>.

To those not connected with dye manufacture it may appear that dye chemistry has lost some of its early importance. This is not to be wondered at when one considers, for example, the change that has taken place in chemistry research in the universities. There was a time when a great number of the foremost teachers of chemistry devoted their efforts to the investigation of dye problems, and the results of their work were published in numerous scientific papers. Today, in the universities, dye chemistry has almost completely given way to new branches of research, e.g. the chemistry of macromolecular compounds and, in particular, research connected with the medical sciences, which naturally arouses much greater public interest. To my knowledge there are only two university chairs of dye chemistry in the western world, one here in Leeds and one in my own home town of Basle. Apart from these, there are only a few scientists in universities who have specifically chosen to work in the field of dyes.

Even in industrial research in the textile field, dye chemistry no longer takes pride of place as it formerly did. It has in fact paved the way for newer lines of research, such as the study of fibres, application research, and the chemistry of auxiliary products and finishing agents.

However, when one considers dye chemistry by itself and not in relation to the other branches of chemical science—which is expanding so rapidly in all directions—it is clear that there is no question of its having lost significance. The contrary is proved by the fact that at present more chemists than ever are active in the search for new Their work, however, attracts very little publicity, as they work in the seclusion of industrial laboratories, and the results of their efforts are published almost exclusively in patent specifications. Such literature is, for the most part, read only by specialists, i.e. by other dye chemists. To my mind this trend is to be deplored, as it is surely most desirable that the new development in dye chemistry should be given greater publicity in the various scientific journals.

The reasons for this continued research activity are the problems continually arising as a result of the great advances being made in the textile field. The more the dye chemist probes into his particular field the more he realises that development is at full spate and also that the results obtained hitherto fall very far short of perfection. You yourselves know that of all the dye ranges available there is not one which can be considered as complete. In some cases certain desirable shades cannot be produced and in others the fastness properties of some brands are not up to the general standard of the range. There is also the desire to have dyes of the same class, possessing similar dyeing properties. In the range of the anthraquinonoid vat dyes, for example—the first member of which was discovered more than fifty years ago—there are no bright scarlets or reds, no bright greenish yellow, and no bright turquoise blue which show the same standard of fastness as the best dyes of this range. We have yet to produce a complete range of dyes which are dischargeable to white and at the same time show the highest degree of fastness. Further, I should like to emphasise that there is still an enormous amount of very important detail work to be done in improving the present ranges of dyes.

After these introductory remarks, I will now endeavour to discuss how the latest developments in the textile field are affecting dye chemistry.

When the dye chemist sets out to determine the most important properties required of dyes, he finds himself confronted with a most difficult problem, which can be solved only by close co-operation with the colourist. A few years ago I had the opportunity of making contact with the dye-consuming industries in several countries in

order to acquaint myself with the application of dyes and to study unsolved problems and trends of development. What impressed me at first was the bewildering complexity that rules every aspect of the application of synthetic dyes. In this connection I have vivid memories of the Conference on The Tinctorial Arts Today organised by your Society at Harrogate in September 1951, which gave such adequate expression to this intriguing variety of problems <sup>2</sup>. In the face of this complexity nothing is more dangerous than the temptation to generalise, but nevertheless the serious student will find general rules which he can follow, or rather to which he will have to cling if he is to avoid losing sight of the essentials.

Let us now discuss the chief properties demanded of a new dye. Apart from the natural need for a commercially usable and, if possible, bright shade together with a reasonable price, there are two main general considerations which the dye chemist must not lose sight of—firstly the need for the best possible fastness properties; and secondly, the need for simplicity, reliability, and economy in application.

#### FASTNESS PROPERTIES

When we survey the various fields of application, we find that the number of articles which have no special requirements regarding fastness to light or wet treatments is steadily decreasing. Everywhere we encounter the demand for faster dyes. We find, too, that users are becoming increasingly "fastness conscious". This tendency represents part of the general endeavour to improve the quality of textiles, particular evidence of which is shown by the creation of new harder-wearing fibres and the increased importance attached to permanent finishes. The logical result of this development is that the fastest types of dyes have gained vastly in importance, and it is not to be wondered at that the dye chemist is devoting more and more of his time to the widening of existing ranges of fast dyes and to the development of new types.

We might now consider the roads open to the chemist in the development of fast dyes. When we examine the nature of fast textile dyeings or prints we see that they are produced with dyes which have to satisfy the following main requirements—

Firstly, the dye should be present on the fibre in as sparingly soluble a form as possible, preferably as an insoluble pigment. In addition to this, it should be so constituted as to bring into play sufficiently strong chemical or physical forces of affinity between the dye and the substrate, this being a factor of decisive importance if the dye still possesses solubilising groups when on the fibre. The word "substrate" in this connection may mean either the fibre itself or a special compound which is either firmly bound to the fibre or incorporated in it and which serves as a link between fibre and dye. These factors constitute the most important prerequisites for good wet-fastness properties, that is to say, insensitiveness to aqueous agents to which the dyed article may be exposed during further processing or wear, such as neutral, acid, or alkaline rinsing, washing, and milling liquors.

In addition, the structure of the dye molecule must be such as to assure good fastness properties which are in no way related to solubility. This refers especially to the highly important property of light fastness. From purely empirical data, the chemist is familiar with a great number of combinations, groupings, components, and substituents which have a favourable influence on light fastness. However, to attempt to extract from this vast collection of facts a set of generally valid rules would be an exceedingly difficult task, as reactions can differ greatly according to the class of dye and type of fibre. One exception is, perhaps, the fact that metal complexes-whether in substance or produced on the fibre-generally lead to dyeings of high fastness to light.

In view of this complicated situation we will assume, when discussing the major principles underlying the structure of fast dyes, that good fastness to light is guaranteed by virtue of the constitution of the dyes concerned. If we start with those types of dyes which do not possess solubilising groups on the fibre and then go on, step by step, to those dyes whose wet fastness properties mainly derive from increased affinity, we can summarise as follows—

- (1) Dyes which are fixed on the fibre via a soluble intermediate form and then reconverted into their original insoluble form. The vat dyes belong to this group, and are still the most important of the fastest textile dyes.
- (2) Insoluble dyes which are synthesised on the fibre. Examples of these are Aniline Black and, in particular, the azoic pigments, which are produced by coupling a diazotised amine with a naphthol, or from corresponding stable compositions of the Rapid Fast and Rapidogen (IG) types. The latest examples are the Phthalogens (FBy)—3-amino-1-imino- $\psi$ -isoindoles, which, along with suitable metallic salts, are developed on the fibre into metal phthalocyanines \*.
- (3) Actual pigments, which can be applied by one of the following methods—
  - (a) By colouring in the dope during the spinning process.
  - (b) By fixation with binding agents, especially those based on synthetic resins. The Aridyes (IC) are examples in this field.
- (4) Dyes whose solubilising groups are split off on the fibre. Examples of these are the sulphuric esters of leuco compounds of vat dyes, as represented by the Indigosols (DH), and the onium dye Alcian Blue (ICI).
- (5) Dyes whose solubilising groups are blocked on the fibre by means of cation-active auxiliaries. This principle can be put to good use in the case of certain direct dyes. An aftertreatment involving the use of copper salts normally has a favourable effect on light fastness.
- (6) Dyes whose solubilising groups are blocked on the fibre by inclusion in a metal-complex

<sup>•</sup> When reference is made to a proprietary brand in order better to particularise a dye group, the designation given by the firm primarily responsible for its development is used.

structure. This principle can be advantageously combined with that of blocking by cation-active compounds. Examples are provided by the polyazo dyes which form stable copper complexes, represented by the Benzo Fast Copper (IG) and Coprantine (Ciba) dyes. The improved fastness of these dyes is, perhaps, based to a certain extent on another principle, which is most strikingly illustrated by the following group (7).

(7) Soluble dyes which are present on the fibre in the form of metal complexes, some active groups of the fibre being incorporated in the complex. This principle has found its chief application in the field of fast wool dyes, e.g. the chrome dyes. It is exemplified especially in the watersoluble premetallised dyes of the Neolan (Ciba) and Palatine Fast (IG) types, and more recently in the Irgalan (Gy) and Cibalan (Ciba) ranges of dyes.

Finally, I should like to mention a principle applied to improve fastness which, although interesting in itself, has lost some of its original significance in modern dye chemistry—

(8) Soluble dyes whose synthesis is completed on the fibre, e.g. by being diazotised and developed with a coupling component or by an after-treatment with a diazotised amine. In general, the principle involved is that of increasing the molecular size of the dye on the fibre, without increasing the number of solubilising groups. A similar principle operates when dye molecules are linked together on the fibre, e.g. by means of an aftertreatment with formaldehyde.

In view of the growing importance of the fastest types of dyes, the dye chemist will give the possibilities just discussed his special attention and modify them further. For example, some of the principles mentioned can, with due modification, be applied to the special class of disperse dyes which are soluble in organic media. Apart from improvement of known methods, there is no doubt that new principles and synthetic means will be found, as shown e.g. by the development of the Carbolans (ICI), where the introduction of long aliphatic chains has increased fastness to wet processing. The suggestion has already been made that dyes should be made which can be polymerised on the fibre or which, by means of actual chemical reactions between dye and substrate, can be bound to the latter by non-polar linkages. Other suggestions refer to the manufacture of pigments in such an exceedingly fine state of division that they could be applied direct from colloidal solutions.

In his search for new fast dyes the chemist endeavours to combine high fastness and brightness in one and the same product. However, when we consider, for example, the field of water-soluble azo dyes, we realise that this problem is by no means an easy one to solve.

When discussing the demand for maximum fastness, we must bear in mind that the conception of fastness is subject to continual change. New fastness requirements appear which were previously unknown. As an example of this I might instance fastness to the various new finishing processes,

based on synthetic resins, such as the anti-crease, anti-shrink, non-felting, and flameproof finishes. New fibres also have brought new fastness problems. For example, it was found that very few blues suitable for dyeing acetate rayon were fast to burnt gas fumes. Some new fully synthetic fibres, when designed for certain purposes, require dyes which will withstand high-temperature setting. finished textiles also are subjected to new processes, which in their turn have an effect on dyes, one example being dry cleaning. In this connection, I should like to remind you of fibre tendering caused by dyes, a problem which was so ably discussed at the 1949 Symposium on Photochemistry in relation to Textiles held by your Society 3. This factor also engages the attention of the dye chemist, and his efforts are at last bearing fruit. We can see, therefore, that the demand for higher fastness itself brings an abundance of problems for the chemist.

#### SIMPLICITY, RELIABILITY, AND ECONOMY IN APPLICATION

It is all too easy for the dye chemist to concentrate on improving fastness properties and neglect other factors such as levelling, penetration, solubility, affinity, and sensitivity to pH, temperature, and salt. What interests him in particular is to what extent a change in the constitution of the dye molecule affects the various easily definable fastness properties. With regard to the demand for simplicity, reliability, and economy in application, the research chemist is apt to consider that, in view of the trouble and patience required to produce a new dye, the dyer or printer might very well exert himself a little and endeavour to apply it successfully, even though its application may be somewhat complicated. However, when one has had the opportunity of acquainting oneself with the problems and difficulties which face the dyer or printer in practical working, it is easy to understand the importance of this demand for simplicity and reliability in application. Industrial dye research has, therefore, directed its attention to this problem, and sees its ideal solution in the production of fast dyes which can be applied in one stage without change in shade. An example of this is found in the latest development in dye chemistry, namely the neutral-dyeing metal complexes-

Apart from the vat dyes for wool, the fastest in the field of wool dyes are the chrome dyes, and efforts to render their application simple, reliable, and economical have, therefore, long been a focal point of interest. These efforts have resulted in the working out of the single-stage chroming process and the development of the metachrome dyes. The premetallised dyes of the Neolan type represent a further important advance along the road to simplicity and reliability. 1952 saw the introduction of two new classes of dyes, the Irgalans and the Cibalans. Both on wool and on nylon, these dyes combine the simplicity of application of the Neolans and the high fastness of the chrome and metachrome dyes. Their fastness can be attributed to the fact that, like the chrome dyes on the fibre, they represent a 1:2 metal complex; i.e. one metal atom is bound to two molecules of dye to form a relatively large complex. The Neolans, on the other hand, are mainly I: I complexes. The new dyes are not only simple and reliable in application and of high fastness, but they are also economical to use on account of their rapid fixation. After the advance in the field of fast wool dyeing represented by the Irgalans and Cibalans, there still remains the urgent need for bright dyes with the same dyeing properties and high standard of fastness. This is a demand which cannot be met fully by the acid anthraquinone dyes, the Carbolans, or the vat dyes for wool—the fastest of the bright wool dyes.

In the field of direct dyes we find a similar though less advanced combination of improved fastness and simplified application. As is well known, the fastness to light and washing of certain direct dyes, which are capable of forming stable complexes, can be improved by coppering after application. The Benzo Fast Copper dyes represented the first special range of this type. The Coprantine dyes applied by the Coprantine process made it possible to produce improved fastness properties by means of a one-bath method. However, the desire for even better fastness led to the development of a more complicated process, namely that of a combined aftertreatment with a copper salt and a cationactive auxiliary product. This development, moreover, offered a wider selection of dyes by permitting the use of fast-to-light direct dyes whose fastness was not permanently improved by coppering alone. This was followed by a step towards simplification in that preparations were introduced which combined a copper salt and a cation-active auxiliary, the first of these being Cuprofix (S). It was then found that these preparations could be applied in combination with a finishing operation, e.g. during anti-creasing, which may allow of further improvement of wet fastness. On the one hand, therefore, we are watching a steady improvement in fastness properties, and on the other a simplification of application procedure.

That the demand for simpler, more reliable, and more economical methods of application is evident in all fields, and that the dye chemist is doing his utmost to meet this demand, can be demonstrated by the following examples—

First of all, we may recall the production of the Duranol (ICI) and the S.R.A. (BrC) dyes, the first disperse dyes, which more than ever are important for their simplicity and reliability in application. It is these dyes which best cover the faults in the newer synthetic fibres, an example being barriness in nylon. Secondly, we may instance the simplified methods of application in the field of azoic dyes represented by the Rapid Fast and Rapidogen compositions, and more recently by the Neutrogens (Fran), which can be developed in neutral steam. The fact that attempts are continually being made to produce improved water-soluble sulphur dyes which are simpler to apply is further evidence of this tendency, and so is the increasing popularity of pigments, which in principle are easy of application. Strenuous efforts are being made to simplify the

application of the vat dyes. These efforts have led to the development of the Indigosols and, more recently, to the special forms of vat dyes which find their application in the modern continuous methods of dyeing.

#### MODERN METHODS OF DYEING

This brings us to the point at which we can discuss the effect which modern methods of dyeing are having on dye chemistry. In this connection interest centres on the new continuous methods of dyeing, such as the pad-steam, Standfast molten metal, hot oil, Uxbridge, and Thermosol methods, and on the various continuous processes for the application of pigments. It is generally recognised that these processes represent a development which will have at least as great an impact on the industry as had the introduction of machine printing. Their main object is to speed up production. This, of course, results in a demand for dyes which can be fixed rapidly. The underlying principle of the above methods may be described as follows—

The dye is applied to the goods in a form in which it has either no affinity or very little affinity for the fibre. This allows complete and even penetration of the goods by the dye suspension or solution. When this has been achieved, fixation is brought about instantaneously, generally by increase of temperature. Subsequent levelling out is no longer possible but also, thanks to the initial penetration, no longer necessary.

The question now is how the dye chemist can do justice to this principle of application, which merely represents a modern form of the chief requirements we have just discussed, viz. simplicity, reliability, and economy in application.

Hitherto, attention has focussed mainly on the vat dyes in the development of new methods of continuous dyeing, and this is hardly to be wondered at in view of the increasing importance of the fastest type of dye and the urgent need for a simplified method of application. As is well known, the continuous application of vat dyes involves applying the dye on the pad, either in pigment form or as a vat acid, neither form having affinity for the fibre, transforming it into a salt of the leuco form which has very high affinity, and then rapidly bringing about fixation by raising the temperature. The need for complete and even penetration and rapid fixation makes necessary the use of vat dyes in a very fine state of division. The manufacture of such dyes, whether as pigments or, perhaps, as stabilised vat acids, represents a highly complex problem, which has not only a chemical but also a physical aspect. If the chemist succeeds in producing dyes of sufficiently small particle size, he is immediately faced with the problem of stabilising the dispersions. As is well known. colloidal systems such as highly divided vat dyes are extremely sensitive to electrolytes and changes in temperature, etc. There is the continual danger of agglomeration. Another problem is how to prevent migration of the pigment dispersion if the goods have to be dried before fixation. Further, the high-speed continuous processes call for vat dyes which allow rapid oxidation and rapid development

during the soaping operation, so that maximum fastness properties may be ensured. In face of the complexity of these problems, it is not surprising that there is only a very small number of vat dyes on the market that meet these various requirements.

While discussing finely divided vat dyes, we should mention modern machine dyeing. As is known, these dyes are of considerable interest in the dyeing of cheeses, cops, beams, and rayon cakes.

In view of the increasing importance of modern continuous processes, the dye chemist will also have to devote his attention to the principle of instantaneous synthesis of dye on the fibre. He will endeavour to develop further the azoics, which represent the most important range in this field, and also to create new products suitable for dye synthesis on the fibre, e.g. the Phthalogens.

Apart from vat dyes present on the fibre in pigment form and pigments synthesised on the fibre, it is the actual pigments themselves which best meet the requirements of continuous methods of application. This applies both to their application by means of synthetic-resin binding agents and to the colouring of man-made fibres in the dope. With regard to pigment preparations which are fixed on the fibre by means of binders, it is quite obvious that they owe their great popularity, particularly in the U.S.A., to their suitability for simple continuous application, and not to their fastness properties, which fall far short of meeting the most stringent requirements. When dealing with pigments or pigment preparations for the dope-colouring of synthetic fibres, the dye chemist will have to pay considerable attention to the fineness and stability of the dispersion. Only pigments in the finest state of division have no adverse effect on the quality of the fibre when applied in the dope.

Soluble dyes, such as the Indigosols, will also be applied to an increasing extent by continuous processes. Therefore, in all fields, the dye chemist is faced with the new task of adapting the dyeing properties of dyes to the new requirements, and, as has already been pointed out, he will have to pay special attention to their physical form. This development means that the assessment of newly discovered dyes cannot be based solely on the requirements of the classic methods of dyeing and fastness tests. It means rather that, in order to ensure a successful continuation of his researches, the dye chemist must be able, in every case, to count on a much more extensive investigation into dyeing properties, which in turn calls for greater recourse to bulk trials.

#### NEW FIBRES

Apart from the new methods of application, the discovery of new fibres will, as already mentioned, also have a far reaching effect on the future development of dye chemistry. Before endeavouring to explain how the dye chemist views this problem of new fibres. I will discuss briefly the present position with regard to the various artificial fibres.

The close similarity between viscose rayon in filament and staple form and the natural cotton fibre explains why no fundamentally new dye problems arose with regard to this regenerated cellulose fibre.

The dyeing of acetate rayon, on the other hand, at first presented a particularly difficult problem, which was solved by the development of the disperse class of dye. However, when one discounts the pigment preparations applied in the dope, there is still a lack of products of high fastness to light and washing suitable for dyeing this pleasing fibre by the more normal methods of application.

The great amount of work carried out on the problem of dyes and dyeing methods for nylon has, in an amazingly short space of time, led to successful results, and we may safely assume that further progress will be made in this field. The main contributions of dye chemistry are the premetallised dyes such as the Perlon Fast (IG) or Vialon Fast (Basf) dyes, the Capracyls (DuP), and the new fast wool dyes, the Irgalans and the Cibalans. When nylon manufacturers succeed in improving the structural uniformity of the nylon filament, we may consider the field of dyes for nylon to have reached a stage of development comparable to that of dyes for wool.

The dyeing of regenerated protein fibres has not given rise to problems for the dye chemist, as these fibres can be dyed in the normal manner with existing wool dyes.

Of an entirely different order, however, are the problems surrounding the dyeing of the latest fully synthetic fibres such as Acrilan, dynel, Orlon, Saran, and Terylene, to mention only those which have been brought to the most advanced stage of development, both technically and commercially. These fibres are based on polyvinyl or polyacrylic compounds or on polyesters, and there is no doubt whatever that by virtue of their outstanding properties, both alone and in conjunction with other fibres, they will secure for themselves a prominent place in the textile industry. In this connection, I should like to remind you of the masterly manner in which Dr. Rowland Hill surveyed this field in the second George Douglas Lecture -- Synthetic Fibres in Prospect and Retrospect 4- and this permits me to confine my discourse to the actual dye problems connected with these fibres.

It is the extremely high resistance of these new synthetic fibres to chemical agents, one of their most valuable properties, which creates a particularly difficult problem for the dye chemist. This resistance accounts for the absence of chemical affinity for existing dyes. In addition to this, their hydrophobic character, limited swelling properties, and smooth surfaces render difficult the penetration and retention of the dye particles by adsorption.

The question is whether, in the face of these rather forbidding obstacles, it will be possible for the dye chemist to solve the dyeing problems involved in the production of new dyes. His task is

further complicated by the fact that the hardwearing nature of these fibres calls for dyes of a correspondingly high standard of fastness.

When we take stock of the various classes of dyes—excluding, for the time being, the latest techniques evolved through research in the application field—we find that the following types of dyes stand the best chances of being selected for dyeing the new fully synthetic fibres.

First choice would fall on the disperse dyes, which contributed so largely to solving the problem of the dyeing of acetate rayon, and which occupy a special place in the dyeing of nylon and Perlon, the first important fully synthetic fibres. As to whether it is possible to produce new disperse dyes which will easily dye fibres such as Orlon and Terylene in shades of sufficiently high fastness is a question which will, for the time being, have to remain unanswered.

The principle of dye synthesis on the fibre also has its interesting aspects. Indeed, the possibility of applying suitable intermediates of small molecular size to the fibre and then building them up into a dye holds out possibilities of technical development.

Colouring these fibres in the dope with the fastest types of pigments or pigment preparations should provide a relatively simple answer to this problem, especially with regard to fastness properties. The fixation of pigments on the surface of the fibre by means of synthetic resins should also offer a satisfactory solution to the problem of dyeing and printing certain styles. This technique should prove especially suitable for the treatment of fibre blends, as it ensures covering in the same shade. The introduction of the synthetic fibres. like the new continuous methods of dyeing, will therefore contribute considerably to furthering the use of pigments in the textile field. It is thus only natural that the dve chemist should also concentrate on the production of very fast pigments and pigment preparations suitable for use on textiles.

However, the classes of dyes just mentioned are by no means suitable for all desired combinations and uses. We have only to consider the enormous number of shades and the numerous possibilities of variation at the different stages in the processing of textile fibres to appreciate this. It is to be sincerely hoped, however, that, despite the technique of dope-colouring and continuous dyeing processes, we shall be able to avoid extreme standardisation and preserve the magic of variety in dyed and printed textiles. It is for this reason that an urgent need exists for the employment of other classes of dyes suitable for dyeing these new types of fibre. What come to mind in this connection are dyes which, like the disperse dyes and pigments already mentioned, do not contain sulphonic acid or carboxyl groups, e.g. vat dyes, vat acids, and basic dyes, or suitable metal complexes. This need constitutes one of the most important current problems in industrial dye research. But, without being unduly pessimistic, I do not believe that this problem can be solved by the dye chemist alone. I believe, in fact, that it is an excellent example of the need for a united

effort by the colourist, the specialist in application research, the dye chemist, the auxiliary products chemist, the engineer, and the fibre chemist. Furthermore, I feel it to be important that, in order to accelerate the solution of dye problems in this field, application and fibre research should take the lead and provide the dyc chemist with the pointers he needs; this view is supported by developments up to date. Much valuable work has already been done. I should like to draw your attention to the work carried out on pressure dyeing at elevated temperatures, to the Thermosol process, the use of swelling agents and carriers, the cuprous ion dyeing method for acrylonitrile fibres, and last but not least to the efforts of fibre chemists to produce polymers which can be more easily dyed. As a result of this work, the dye chemist can press forward on a broader front, that is to say, he can include in his researches other types of dyes, as for example dyes suitable for animal fibres. However, a great deal remains to be done, especially with regard to a thorough investigation of the results gained so far. On such a basis it will be easier for the dye chemist to evolve new products suitable for the dyeing of the new fibres.

#### CONCLUSION

If we survey the demands made on new dyes as a result of modern developments in the textile field, we realise that the dye chemist is faced with an increasing number of unsolved problems. I have tried in this lecture to portray for you the factors which influence his approach to these problems and in particular to discuss the posibilities of solving them by chemical means. I have deliberately avoided indulging in speculative forecasts. A close scrutiny of the development of dye chemistry shows that progress has seldom been due to flashes of inspiration, but rather to dogged and purposeful research carried on by the experienced chemist. Another element is very often that of chance, which, however, favours only him who is capable of unerring observation and who is ready to investigate the true nature of his discovery.

I should like to stress once again that discussion of the various problems has shown that a practical solution is to be found only in the close cooperation of all branches of research related to the textile field. But I feel that a decisive factor in ensuring continued progress is that none of the branches of research should be considered subordinate to the others, and that each should view the production of something essentially new as its prime task. For the dye chemist this means not only the production of new dyes but also the discovery of new types of dyes. He can do justice to this task only by utilising to the full the latest discoveries and methods, and especially by a study of advances made in the fields of plastics, physical chemistry, and physics. Fresh impetus can be given to dye chemistry also by the use of newly discovered starting materials such as the polycyclic compounds which can be isolated from coal tar, or of the new products now made available by advances in the chemistry and technology of mineral oils. If the dye chemist is then successful in finding something new which promises to be of value, a means can always be found to put it to practical use. One of the best examples of this is the discovery and development of the phthalocyanines, the newest of the important chromogens. This work led to the introduction of the new Alcian Blue and Phthalogen types, and to dyes which can be included in the classic vat, sulphur, and direct

ranges

Apart from the search for new and better dyes, there is a need for increased fundamental research, which is essential if further progress in the field of dye chemistry is to continue. On examining the present state of these more theoretical studies, one finds that much still remains to be explored. I need instance only the great quantity of data accumulated over the past hundred years by industrial dye chemists. These have now to be sifted in the light of modern theoretical organic chemistry with a view to obtaining a better understanding of the nature of colour, or of the relationship between constitution on the one hand, and colour, fastness, and dyeing properties on the other. I doubt very much whether the industrial chemist will be able to solve these problems unaided, faced as he is with other problems demanding immediate solution. The atmosphere necessarily prevailing in the laboratories of competitive enterprises is hardly conducive to the pursuit of fundamental theoretical research. It is in the interest of progress that dve chemistry be accorded greater importance in the universities. This has always been recognised by far-sighted men in the industry, and it is to be hoped that their efforts in furthering this will bear

In conclusion, my best thanks are due to all those who have assisted in the preparation of this paper. Moreover, may I be allowed, as a dye chemist, to express my appreciation of the help given to all workers in the field of industrial dye chemistry by specialists in application research, by textile chemists, and particularly by the technical and practical men in dyehouses and printworks. It is this spirit of close co-operation which enables the dye chemist to produce new dyes of value to industry.

(MS, received 13th April 1953)

#### References

Mr. P. A. Holt, in proposing a hearty vote of thanks to Dr. Holbro, said that the lecture had been one after George Douglas's own heart—a striving for improvement, a reach to greater heights of endeavour.

The dyeing industry is turning more and more to continuous methods of dyeing as opposed to batch methods. Printers and bleachers have, of course, long used such methods. Continuous processing should tend to give us work of much better quality and consistency, and at the same time be cheaper in labour costs and overheads. A few years ago we never imagined that we should be vat-dyeing cotton or rayon fabrics at the rate of 50 yd./min., or

1000 yd. in 20 min. Yet this is possible today by the Standfast molten metal process and the du Pont pad-steam process. What is also important is that both of these processes deal efficiently with short runs of cloth, very little time being taken in cleaning down between runs.

Continuous pad dyeing of spun viscose rayon with direct dyes produces results of a levelness far surpassing that obtained by the more conventional methods, where the uptake of the dye by the fibre continued over a period of some hours and where preferential absorption of dye emphasised yarn or weave variations. Continuous dyeing of acetate rayon has been practised for some years now in the U.S.A. and Canada. Pigment compositions which can be applied mechanically are also being developed. The dyemaker should treat this aspect of dyeing very seriously. Instead of allowing the fabric to feed on the dye, the fabric must be forcibly fed with the exact amount of dye required.

Speaking in the West Riding, one cannot ignore wool, and whilst there has not, perhaps, been the same attention paid to continuous dyeing methods for wool fabrics, of all fibres wool is probably the most affected by variations in the times of batch

processing.

The consumer will pay no more for fabric dyed by a new technique unless, of course, it offers greater value. At present the economies made in labour costs and overheads by continuous processing are somewhat offset by the process being more expensive in the cost of dye used. This conception of continuous dyeing is, of course, as great a challenge to the dyer as to the dyemaker, for the dyer will have to alter completely his ideas of colour matching. The main point seems to be that we should make our search for new dyes completely untrammelled by tradition.

Speaking as a dye user, Mr. Holt assured Dr. Holbro as a dye manufacturer that he would find the dyers in this country very responsive and willing to help in the search for new dyes in relation

to modern requirements.

Professor W. Bradley, in seconding the vote of thanks, said that Dr. Holbro came of a long line of eminent Swiss scientists and technologists, who had done a great deal to promote the wellbeing, not only of their homeland, but of science and technology throughout the world. One thought particularly of Alfred Werner, the discoverer of the principle of co-ordination; Traugott Sandmeyer, whose synthesis of indigo ranked amongst the most elegant yet devised, and whose methods of replacing aromatic amino groups by halogens and the cyano group were known to every young organic chemist; and G. Engi, the first to learn the art of brominating indigo.

Dr. Holbro himself began his research life as an electrochemist in the laboratory of Professor F. Fichter. His task, which he completed, was to ascertain the features of structure which limited the application of Kolbe's synthesis to salts of unsaturated aliphatic acids, and anyone who has engaged in work of this kind, or has seen it performed, will know the skill and patience which are required for success.

<sup>&</sup>lt;sup>1</sup> Levinstein, H., J.S.D.C., 65, 269 (1949).

<sup>&</sup>lt;sup>2</sup> Ibid., 67, 489-635 (Dec. 1951).

<sup>&</sup>lt;sup>3</sup> Ibid., 65, 585-788 (Dec. 1949).

Hill, R., ibid., 68, 158 (May 1952).

It was only to be expected that such ability as Dr. Holbro had shown would be recognised by the universities or industry. In fact Dr. Holbro joined the Ciba organisation, and during the past fifteen years his progress has been marked by the appearance of a succession of patents, fourteen of them British.

A few years after joining Ciba as assistant to Dr. R. Tobler, Dr. Holbro turned to typical organic chemical interests, including the chemistry

of the higher hydrocarbons of coal tar. Fluoranthene gives a dibromo derivative under ordinary conditions of bromination in nitrobenzene or carbon disulphide, but the constitution of the product was not known. Fluoranthene contains ten replaceable atoms of hydrogen, and twenty-two forms of dibromo derivative are possible. Dr. Holbro correctly ascertained the constitution of the product of direct bromination, and confirmed his conclusion by an elegant synthesis.

#### COMMUNICATION

#### Studies in the Fundamental Processes of Textile Printing V—The Transfer of Disperse and Water-soluble Dyes to Cellulose Acetate during Steaming

E. H. DARUWALLA and H. A. TURNER

The experimental methods used in the preceding studies in this series <sup>1-4</sup> have been adapted to the investigation of the transfer of selected disperse and soluble acetate rayon dyes to cellulose acetate during steaming. The effects of concentration, chemical structure, and particle size of the dye and of the presence of electrolytes and swelling agents for the fibre substance have been examined for appropriate conditions of application. The results are discussed with special reference to corresponding details of the dyeing behaviour as revealed by quantitative studies of other workers. In many of these details there is an unusually close correspondence between dyeing behaviour and printing transfer.

#### Introduction

OBJECTS OF THE INVESTIGATION

Up to the present, in this series of investigations, the thickening agent has been represented predominantly by starch 1-4, and the fibre substance exclusively by cellulose. It was desired to extend the study to fibre substances the properties of which were quite different from those of cellulose. and, for this purpose, cellulose acetate appeared prospectively to be one of the most interesting. Its dyeing behaviour is different from that of any of the usual natural fibres and many of the manufactured ones, generally requiring the use of different classes of dyes. Its capacity for taking up water is much lower than that of any of the normal cellulosic and protein fibres. Previous work 4, while it has not always been able to define the factor precisely, has shown that the distribution of water between the thickener and the fibre substance, during drying and steaming, has a great influence upon the consequent distribution of dye between the two phases. In a starchcellulose acetate system, therefore, where water distribution is expected to be quite different from that in systems previously examined, there should be different dye-transfer behaviour.

There have been a number of attempts to define quantitatively the adsorption, from an aqueous medium, of a variety of dyes and of simpler organic compounds, regarded as analogues of dyes, by celluses acetate. These have included both water-soluble and water-insoluble compounds 5-22. There does not appear to have been any comparable study for application by printing, although practical printing trials of one kind or another have been numerous. In recent years, much work has been directed towards improving the receptivity of the fibre for dyes, mainly of those classes in which the normal adsorption is low. Agents have been

used, as additions to dyebaths and printing pastes, whose action is essentially to swell the fibre.

The present work is based on a technique first described by Munshi and Turner 1, in which a printing system before steaming is represented by a film of the dry thickening agent, containing the dye and other required additions, maintained in an atmosphere of steam in intimate contact with a film of the fibre substance. At the end of a predetermined time the proportion of dye which has diffused from the thickener to the fibresubstance is measured. It will be seen that, when this method is applied to systems in which the fibre-substance is cellulose acetate, certain technical problems, associated with the printing of fabrics made from cellulose acetate rayons, are ignored. An important one is presented by the difficulty of securing, at the moment of application, rapid and complete penetration of the printing paste into the interstices of the fabric, owing to the incomplete wetting of the fibre surfaces by water and aqueous solutions.

#### SUMMARY OF PRINCIPAL RESULTS

- (1) When a film of cellulose acetate, of a composition identical with that used for the manufacture of normal acetate rayon, is maintained in steam for a short period it becomes delustred. A similar film steamed in contact with a film of starch retains its transparency for several hours, showing that the starch has markedly diminished the ability of the cellulose acetate to retain the moisture taken up from the steam.
- (2) The general characteristics of the transfer of disperse and soluble acetate rayon dyes from starch to cellulose acetate are, respectively, closely similar to those of their transfer from water to the acetate during dyeing.

(3) The exhaustion of the disperse dyes from the thickener to the cellulose acetate at equilibrium is high, being of the order of 90% in the dyes examined. For a given dye it is independent, over a wide range, of the initial concentration of dye in the thickener layer. The higher the initial concentration of dye in the thickener, the longer is the time required to reach equilibrium conditions.

(4) Following the observation in (3) above, a detailed examination of the transfer behaviour of Duranol Red 2B (ICI) showed that a constant partition of the dye at equilibrium was maintained with increasing proportions of dye in the system until the saturation value of the dye in the acetate was reached. The attainment of saturation con-

ditions is sharply defined.

(5) Progressive reduction in the particle size of Duranol Red 2B, brought about by grinding in a ball-mill under standard conditions for increasing lengths of time, increased the rate of transfer of the dye to the cellulose acetate. Where equilibrium could be attained, the time of grinding did not affect the value for the final exhaustion. In experiments with coarsely divided dye, transfer was so slow that equilibrium conditions could not be reached, but the indication was that the equilibrium transfer would be the same as with finer dispersions. After 20 hr., further grinding had no effect either on the rate of transfer or on the value for equilibrium exhaustion.

(6) Addition of neutral electrolyte (sodium chloride) to the thickener phase caused no alteration in the equilibrium partition ratio for Duranol Red 2B between starch and cellulose acetate.

(7) Desorption experiments were performed in which a film of cellulose acetate containing a disperse dye was steamed in contact with a film of

plain starch. In these experiments—

(a) The rate of desorption (quantity of dye transferred in unit time across unit area of the surface of separation between the phases) was small compared with the corresponding rate of adsorption, because the proportion of the total dye which migrated was much smaller also.

(b) Equilibrium was reached in about the same time both for adsorption and for desorption.

(c) At the respective equilibria, the proportion of the total dye present in the cellulose acetate film was greater for the approach by desorption than for the approach by adsorption.

(d) The distribution of dye between the two phases at equilibrium was independent of the manner in which the dye had first been introduced into the cellulose acetate, viz. by dissolving it in the acetone solution of the acetate from which the film was made, or by transferring it from a dyecontaining starch film in the usual steaming procedure.

(8) An increase in length of the terminal hydrocarbon chain R of the disperse dyes—

from  $C_2H_5$  to  $C_6H_{13}$  decreased the rate of transfer from starch to cellulose acetate, so that equilibrium conditions could not be attained in 8 hr. steaming. (9) Where possible, each of the soluble dyes\* investigated was chosen to be a derivative (sodium sulphuric ester) of a disperse dye the transfer properties of which had also been studied. The exhaustion of the soluble dye during the whole course of steaming was less than that of the corresponding disperse dye, and in no case had definite equilibrium conditions been established at the end of 8 hr. steaming.

(10) The transfer of soluble dyes\* to the cellulose acetate was, in general, increased by the addition of neutral electrolyte to the printing paste, the specific increase being different for different dyes.

(11) When a wet starch paste containing a soluble dye\* was spread in a uniform thin layer over the surface of a film of cellulose acetate, and dried in air at 18–50°c., there was a marked transfer of dye to the acetate film. If the combined films were then steamed, some dye returned to the starch layer during the first hour; but with more prolonged steaming there was a net transfer to the acetate. On examining cross-sections of the films, it was found that the dye had penetrated into the acetate only for a short distance during the drying stage. On steaming, no further penetration could be observed for the first one to two hours; thereafter the dye slowly diffused further into the acetate film.

(12) The change in constitution of the soluble dye—

$$NO_2 \longrightarrow N:N \longrightarrow -NR\cdot CH_2 \cdot CH_2 \cdot O \cdot SO_2 Na$$

from  $R=C_2H_5$  to  $R=C_0H_{13}$  caused little difference in the exhaustion of the pure dye, but a specified addition of sodium chloride brought about a relatively greater increase in the exhaustion of the second dye.

(13) The disperse dyes investigated remained in the particulate condition when incorporated in a starch paste which was then dried at room temperature. A cross-section of this film showed only a faint coloration. After steaming for a short period (15–30 min.) the degree of dispersion within the starch was greatly increased, and this was indicated by the development of an intense colour.

(14) Each disperse dye and its corresponding soluble derivative were destroyed during steaming when sodium alginate was used as a thickener, the soluble form being the more rapidly decolorised.

#### Experimental

MATERIALS

Thyes

We are much indebted to Imperial Chemical Industries Ltd., Dyestuffs Division, for the supply of dyes and intermediates, and for information which has facilitated the preparation and purification of many of the products mentioned below.

DURANGL RED 2B (ICI)-



 In this paper the term soluble dues refers only to soluble dues of the type R O SO<sub>8</sub>Na specially designed for the dyeling and printing of normal cellulose acetate rayon. The commercial product was freed from electrolytes and dispersing agents by repeated extraction with boiling water. The product was then dried at 60°c., crystallised three times from acetone solution, and dried first in the oven at 60°c. and then over phosphorus pentoxide. After the second crystallisation, the melting point remained constant at 200°c. (uncorr.) and the optical density of a standard solution in acetone remained unchanged.

DISPERSOL FAST SCARLET B (ICI) 23-

$$NO_3 \underbrace{\hspace{1cm}} -N;N \underbrace{\hspace{1cm}} -N \underbrace{\hspace{1cm}}$$

Purified p-nitroaniline (m.p. 148°c.) was dissolved in 10 N. hydrochloric acid, diluted with water, and diazotised by the usual procedure. It was then coupled with a suspension of N-ethyl-N- $\beta$ -hydroxy-ethylaniline hydrochloride in excess aqueous hydrochloric acid, until tests with alkaline H acid solution and diazotised p-nitroaniline showed the completion of the reaction. The red precipitate was washed at the pump with distilled water, dried at  $60^{\circ}$ c., and crystallised repeatedly from acetone solution until the melting point remained constant ( $160^{\circ}$ c.) and the optical density in standard acetone solution was unchanged.

This dye was prepared by coupling N-n-hexyl-N- $\beta$ -hydroxyethylaniline with diazotised purified p-nitroaniline  $^{24}$ . After isolation, washing, and drying, the product was crystallised four times from iso-propyl alcohol until the m.p. (109°C., uncorr.) and the optical density in standard acetone solution remained unchanged.

Solacet Fast Crimson B (ICI)—The commercial dye was first washed at the pump with several changes of cold, distilled water, dried, and crystallised repeatedly from acetone solution until the optical density in standard acetone solution remained unchanged. In drying the pure product a temperature of 60°c. was not exceeded.

SOLACET FAST SCARLET B (ICI) 22-

$$NO_2 \\ \hline \\ -N:N- \\ \hline \\ -N \\ \hline \\ CH_4\cdot CH_4\cdot O\cdot 8O_3Na$$

This dye was prepared by coupling diazotised p-nitroaniline with sodium  $\beta$ -N-ethylanilinoethyl sulphate  $^{15}$ . A pure specimen of the sulphuric ester was provided by ICI. The sodium salt was prepared by suspending the ester in water and adding 10 N. sodium hydroxide slowly with stirring until complete solution resulted and a permanent faint pink coloration was obtained. To this solution, containing 27 g. of the ester, 27 g. of anhydrous sodium carbonate was added. Sodium chloride (35 g.) was added, and the solution cooled. Contrary to expectation, it was found impossible to induce separation of the sulphate by salting out, and it was therefore necessary to use the solution

directly for coupling with the diazonium solution, which occupied 1 hr. This calls for no special comment, except that it was found safe to keep the diazotisation mixture in the cold for a further 1 hr. to complete formation of the dye, which was then filtered off, washed with water, and crystallised repeatedly from acetone solution until the optical density in standard acetone solution remained unchanged.

Soluble Dye A\*—
$$NO_{z} \longrightarrow N:N \longrightarrow N \xrightarrow{C_{0}H_{13}} CH_{z}\cdot O\cdot SO_{3}Na$$

This was prepared by coupling diazotised p-nitroaniline with sodium  $\beta$ -N-n-hexylanilinoethyl sulphate  $^{24}$ . The latter, as supplied, was not a pure specimen, and, since it is somewhat unstable, it was not further purified before use, but was titrated with a standard solution of diazotised p-nitroaniline so that the quantities of reactants necessary could be calculated. Diazotisation and coupling were carried out in the usual way, and, after isolation, the resultant dye was purified by repeated crystallisation from a solution in equal proportions of acetone and isopropyl alcohol, with a final crystallisation from isopropyl alcohol alone.

#### Thickeners

Starch  $^{1,2}$ —A high-grade farina of low ash content (0.28% after ignition with concentrated sulphuric acid).

Sodium Alginate 1,2—Foodstuff grade, highviscosity (Alginate Industries Ltd.).

#### Assistants

SODIUM CHLORIDE (BDH)- AnalaR.

DIETHYLENE GLYCOL (BDH), ETHYL LACTATE (BDH), SODIUM THIOCYANATE (Johnson)— All "chemically pure".

Sodium NN-Dibenzylsulphanilate—Solution Salt SV (ICI), a commercial product.

#### Fibre Substance

For the supply and characterisation of the raw material for making cellulose acetate films, and for many helpful hints on the preparation of these films, we are greatly indebted to Dr. F. Howlett and Lansil Ltd., Lancaster.

Cellulose Acetate—Secondary cellulose acetate in fibre form, as for the manufacture of normal acetate rayon, with the following characteristics—

A 10% (wt./vol.) solution of the cellulose acetate in acetone was prepared by thorough shaking followed by standing overnight. The solution was very faintly cloudy. This was spread, as described later, in a thin uniform layer on a carefully levelled glass plate. During the subsequent removal of the solvent, the layer was protected from dust, and evaporation was allowed to take place into a dry atmosphere at room temperature in order to ensure

<sup>.</sup> This dye is not directly associated with a maker's brand name, and has been given the present name for convenience.

that the resulting film should be clear. It is essential that the concentration of the original solution shall not fall far below the specified 10%, otherwise the formation of cloudy films is to be expected. After being dried overnight, the film was detached from the plate, maintained at 40°c, for 4 hr. to assist in removal of residual acetone, and then tested for thickness, the portions in which the thickness lay between 0.0035 and 0.0040 cm. being retained for use. The last traces of acetone were removed by soaking in changes of cold distilled water until no coloration was obtained when a dry mixture of anhydrous sodium nitroprusside (1%), anhydrous sodium carbonate (49.5%), and anhydrous ammonium sulphate (49.5%) was added to the washings 27. After being washed in this manner and then dried to constant weight at 110°C., one disc of the film, Hin. in diameter, weighed 0.0103 g. The spreading of the acetate film was carried out with an apparatus which consisted of two stout brass end members cubical in shape, joined by a rigid brass strip. To this strip a stainless steel doctor, with the lower edge ground accurately linear, was clamped by means of thumbscrews. This apparatus could therefore slide along the glass plate upon which it rested, spreading a layer of dope which was supplied in excess at one side of the doctor. The clearance between the doctor blade and the plate was adjusted at two points by means of feeler gauges.

Cellulose Film—Ordinary non waterproof Cellophane, 300 PT grade, was used. It was freed from glycerol by prolonged soaking in several changes of water until required. One disc, 116 in. in diameter, after soaking and drying to constant

weight at 110°c., weighed 0.0052 g.

#### Dye Suspensions

Fine suspensions of the insoluble acetate rayon dyes were made from the purified crystalline material by grinding with distilled water in a small ball-mill. This consisted essentially of a cylindrical glass weighing bottle, of capacity 50 ml., disposed with its long axis horizontal and rotated about this axis at a constant speed of 240 r.p.m. It contained about 200 stainless steel ball bearings ( $\frac{3}{16}$  in. in diameter). For a single grinding 0·25 g. of dye was placed in the mill along with 25 ml. of water, ground for the desired time, and then washed out and made up to 250 ml. with water. This formed the standard stock suspension.

#### PREPARATION OF THE DYE-CONTAINING STARCH PASTE

Details of the preparation have already been given <sup>1</sup>, and little further comment is called for. Ground and sieved farina (10 g.) was suspended in a total volume of 150 ml. of water. The temperature of the jacket round the cooking vessel was kept at 95°c., heating of the paste being continued for 30 min. after gelatinisation. The paste was allowed to cool with continued stirring to 35°c., when it was used at once for spreading.

#### SPREADING OF THE PASTE

The normal procedure in former dye-transfer experiments has been to spread the dye-containing

paste in a layer of standard thickness upon a glass plate, to dry this down in the cold to a solid film, and then to attach specified areas of this solid film to a film of the fibre substance. This method was unsatisfactory with cellulose acetate, because ready and permanent adhesion could not be obtained. A modification of the procedure, which had already been used in some of the former investigations 2-4 was therefore adopted. Cellulose acetate film which had been soaked in water was taken, excess water removed by blotting, and the film then pressed firmly and evenly into contact with a glass plate. A uniform layer of the starch paste was then spread over this film, and the whole, supported on the plate, was dried in a gentle current of air at room temperature, care being taken that the glass plate was supported in a strictly horizontal position, so that any further flow of the starch was avoided. The humidity of the air was regulated, and for most of the work it was maintained at 75% R.H. If the drying is allowed to take place too rapidly, cracking of the starch film or separation from the acetate may result. A lower R.H. than 75% is needed if high concentrations of electrolytes, especially deliquescents, are incorporated in the starch paste. The composite film of cellulose acetate and starch was normally preserved over saturated sodium chloride solution until required for use.

During the drying process, some dye was transferred from the starch to the cellulose acetate film. With disperse dyes, the proportion was normally very small; with the soluble dyes, it was appreciable, Before a preparation was used in steaming experiments, a representative sample was taken, and the proportion of dye transferred was determined.

#### STEAMING

The steaming apparatus and the procedure have already been described in detail <sup>1,2</sup>.

#### ESTIMATION OF DYE TRANSFERRED

After being steamed for the desired time, the composite film was removed from the steamer. Samples of standard area were cut from this by means of a circular punch. The starch was separated from the cellulose acetate, and the dye in the former extracted by standing overnight in the cold with a known volume of 20% aqueous pyridine solution. The acetate film was dissolved completely in a known volume of dry acetone. The dye in each solution was estimated absorptiometrically on a normal Spekker (Hilger) instrument, which was first calibrated with standard solutions of the pure dye in the appropriate solvent. In any one experiment, the total dye present in the system was calculated by adding together the separately estimated dye contents of the constituent films. Results were generally expressed as-

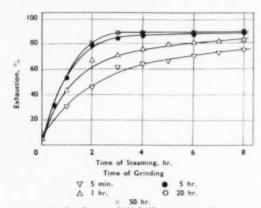
% Exhaustion =  $\frac{\text{(Wt. of dye in acetate)} \times 100}{\text{(Total wt. of dye in system)}}$ 

#### Results

#### EFFECT OF GRINDING ON DYE TRANSFER

Duranol Red 2B was the dye used in this series of experiments. The purified, crystalline material was ground as described for different periods in distilled water, and then incorporated in a standard proportion of a starch paste and used for transfer determinations. It will be seen (Fig. 1) that—

(a) Transfer on steaming is appreciable even with the coarsely crystalline dye which has been ground for 5 min. only, a proceeding which does little more



Initial Dye Content 0-131-0-137 mg. per unit disc Fig. 1— Effect of Grinding upon Transfer of Pure Duranol Red 2B from Starch to Cellulose Acetate upon Steaming

than break up groups of crystals. Transfer in this case is very uneven, the dye in the cellulose acetate being found in patches, corresponding chiefly to the presence of large dye particles in the starch film. As steaming continues, active dispersion within the starch takes place: this was shown by the development of a red coloration throughout the starch film.

(b) As the time of grinding lengthens, and the degree of comminution increases, the rate of transfer also increases and the time necessary to reach equilibrium is diminished. For the conditions which give the slower transfer, the indications are that the same equilibrium exhaustion value would be reached if the steaming could be continued long enough.

(c) Direct determinations of particle size have not been attempted, but the experiments show that grinding for longer than 20 hr. has no further effect upon the transfer behaviour. This probably means that the limits of fineness in the suspension attainable by the use of a ball-mill have been reached.

(d) The reproducibility of the experimental results, in so far as they are affected by the time of preliminary grinding, is shown to be satisfactory by the agreement in repeat experiments with fresh dispersions (Table I).

Table I

Transfer of Pure Duranol Red 2B
(Dye (mg.) per unit area of contact, starch-acetate)

Time of	Present in	Transferred to Acetate during		
Grinding	Complete	Steaming to Equilibrium		
(hr.)	System, before Steaming	Original Experiment	Repeat Experiments	
20	0·135	0·121	0·123, 0·119	
	0·130	0·042	0·041, 0·043	
50	0·132	0·117	0.116, 0.119	
	0·134	0·121	0.122, 0.123	

POSSIBILITY OF LOSS OF DYE DURING STEAMING

Most of the disperse dyes for cellulose acetate tend to sublime on heating. In addition, for those which contain the azo group in the molecule, the possibility of reduction by the starch during prolonged steaming cannot be ignored. To investigate these possibilities, the total dye content in the starch–cellulose acetate system was determined before and after prolonged steaming. The agreement (Table II) is well within the limits of experimental error.

TABLE II

Dye	Time of Steaming (hr.)	Dye in Starch + Dye in Acetate (mg. per unit area of contact)
Duranol Red 2B	Nil 8	0·167 0·168
Dispersol Fast Scarlet B	Nil 8	0·136 0·135
Disperse Dye A	Nil 8	0.0266 $0.0264$
Solacet Fast Crimson B	Nil	0-077 0-076
Solacet Fast Scarlet B	Nil 8	0·081 0·082
Soluble Dye A	Nil 8	$0.254 \\ 0.254$

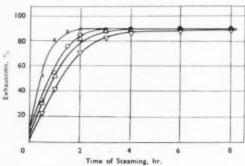
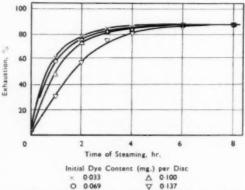


Fig. 2 — Effect of Initial Concentration of Pure Duranol Red 2B in Starch on Transfer to Cellulose Acetate during Steaming



O 0069 
V 0137

Fig. 3 — Effect of Initial Concentration of Pure Dispersol Fast Scarlet B in Starch on Transfer to Cellulose Acetate during Steaming

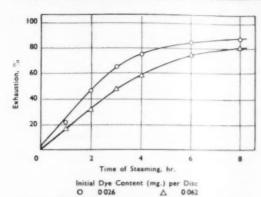
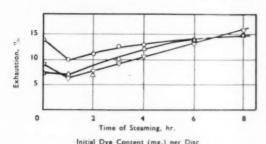


Fig. 4— Effect of Initial Concentration of Pure Disperse Dye A in Starch upon Transfer to Cellulose Acetate during Steaming



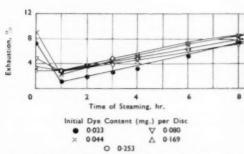
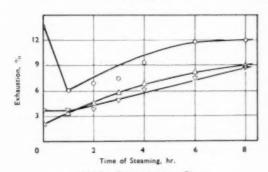


Fig. 6.— Effect of Initial Concentration of Pure Solacet Fast Scarlet B in Starch upon Transfer to Cellulose Acetate during Steaming



Initial Dye Content (mg.) per Disc

O 0021

V 0000

£ 0 0255

Fig. 7— Effect of Initial Concentration of Pure Soluble Dye A
in Starch upon Transfer to Cellulose Acetate during Steaming

#### EFFECT OF INITIAL DYE CONCENTRATION IN STARCH ON EXHAUSTION

The results of experiments with Duranol Red 2B, Dispersol Fast Scarlet B, Disperse Dye A, Solacet Fast Crimson B, Solacet Fast Scarlet B, and Soluble Dye A are shown in Fig. 2–7 inclusive. With the disperse dyes the time of grinding was always 20 hr.

#### Disperse Dyes

The following general conclusions apply for all the disperse dyes examined—

(a) There is a small exhaustion of dye which occurs when the starch paste is being dried in the cold in contact with the cellulose acetate. It is proportionately greater as the amount of dye in the system is less, and ranges from about 1 to 10%. It is generally higher for Duranol Red 2B than for the other two dyes.

(b) The relative rate of exhaustion for any one dye is greater, and the time required to reach equilibrium is less, as the initial concentration of dye in the starch film is diminished.

(c) Where equilibrium is attained, the exhaustion is independent of the initial concentration of dye in the starch film until a certain initial concentration is exceeded. The slow transfer of Disperse Dye A made attainment of equilibrium impossible under the experimental conditions employed.

DURANOL RED 2B— From the results in Fig. 2 and from additional determinations, an adsorption isotherm, relating the proportion of dye in the fibre substance at equilibrium to the proportion of dye remaining in the starch, has been constructed, and is shown in Fig. 8. It will be seen that, over a

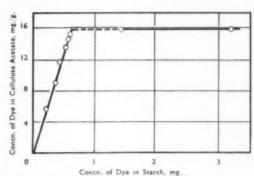


Fig. 8— Isotherm for Distribution of Pure Duranol Red 2B between Starch and Cellulose Acetate during Steaming

considerable range of concentration, there is a constant partition between the two phases, which is terminated abruptly by the attainment of saturation in the acetate phase. Special attention is drawn to this feature, which has an important bearing on the mechanism of dye transfer, and which will be discussed later (p. 251).

DISPERSOL FAST SCARLET B— A similar isotherm is given (Fig. 9) during the transfer of this dye, though here the saturation value for the cellulose

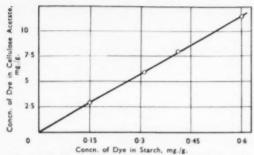


Fig. 9—Isotherm for Distribution of Pure Dispersol Fast Scarlet B between Starch and Cellulose Acetate during Steaming

acetate has not been attained. Transfer experiments with these high concentrations of dye are laborious, and require very long steaming times to ensure that true equilibrium is established.

#### Soluble Dyes

There is a general similarity of transfer behaviour for the three examples investigated (Fig. 5–7), and the following features are common to all—

(a) For the pure dyes without assistant, the level of exhaustion to the acetate is low, and has nowhere exceeded 16%.

(b) The transfer during the drying-on of the starch paste in the cold is important. It may be equal to, or even greater than, the exhaustion produced at any subsequent time during the steaming.

(c) For the first hour of steaming, the exhaustion either remains substantially constant or else diminishes, i.e. there is a reverse migration of dye to the thickener. For longer times of steaming, the exhaustion slowly increases as steaming progresses, but with none of the dyes is there a definite establishment of equilibrium conditions in a total time of 8 hr. In many of the experiments it is plain that a much longer period would be required for this to take place. It has been shown in earlier investigations <sup>1</sup> that steaming times of more than 8 hr. with the present type of systems are not very practical, since degradation and liquefaction of the thickener layer may take place.

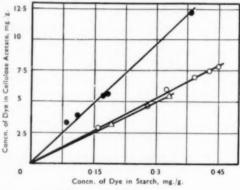
(d) The effect upon transfer behaviour of initial dye concentration in the thickener layer is complex. The exhaustion on drying alone and the drop in exhaustion during the first hour of steaming are both greater the lower the initial concentration. This effect is especially pronounced with Soluble Dye A (Fig. 7). On the whole, relatively high exhaustion also accompanies low initial concentration after more prolonged steaming, but this order may be changed as the time of steaming increases still further. The effects of initial concentration in modifying the transfer behaviour with Soluble Dye A, which has a large hydrocarbon addendum, are, as already noted, very pronounced. At first sight they suggest that, when the dye is presented much diluted to the cellulose acetate, it is very mobile both in the cold and at the temperature of the steamer. For higher concentrations, its mobility seems to be below that of the other Solacet dyes in the cold and at higher temperatures

EFFECT OF SODIUM CHLORIDE UPON EXHAUSTION

The effect of additions of sodium chloride to the starch paste, along with the dye, was investigated for a representative disperse dye— Duranol Red 2B— and for the three soluble dyes.

#### Duranol Red 2B

Quite apart from the fact that the disperse dye is applied under conditions which do not favour ionisation of any of the potentially ionic groups, any substantial increase in the exhaustion of the dye cannot be expected if salt is added, since the exhaustion at equilibrium in its absence is almost complete. The isotherms for Duranol Red 2B without salt and with a constant content of 0·136 mg. of sodium chloride per unit disc are compared in Fig. 10, and it is seen that they agree



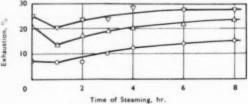
- Desorption without salt
- O Adsorption without salt
- Adsorption with salt

Fig. 10—Adsorption and Desorption Isotherms of Duranol Red 2B

almost within the limits of experimental error, though with an indication that the presence of salt will diminish rather than increase the exhaustion of the dye.

#### Soluble Dyes

From Fig. 11–13 it is seen that the presence of sodium chloride along with a soluble dye has the invariable effect of increasing the exhaustion of the dye during the whole course of the transfer during steaming. The specific effect of a given proportion



Initial Dye Content (C) = 0.058 mg. per disc Salt Content

> O − Δ 10 C ∇ 20 C

Fig. 11— Effect of Sodium Chloride upon Transfer of Pure Solacet Fast Crimson B from Starch to Cellulose Acetate during Steaming

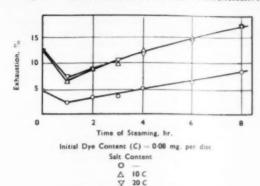


FIG. 12—Effect of Sodium Chloride upon Transfer of Pure Solacet Fast Scarlet B from Starch to Cellulose Acctate during Steaming

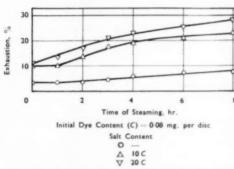


Fig. 13— Effect of Sodium Chloride upon Transfer of Pure Soluble Dve A from Starch to Cellulose Acetate during Steaming

of salt varies from dye to dye. The general shape of the transfer curves is not altered. With some dyes, e.g. Solacet Fast Scarlet B (Fig. 12), certain proportions of salt accentuate the reverse transfer of dye during the first hour of steaming; with others, e.g. Soluble Dye A (Fig. 13), they eliminate it.

#### EFFECT OF CHANGES IN CONDITIONS OF DRYING DOWN

For acid dyes in comparable systems, a suggestion has been made 4 that dyes which have a low affinity for both phases are controlled in their distribution by the proportion of total water in each of the phases of the system. Assuming this to be valid for the present components, it was thought that the substantial transfer of dye to the cellulose acetate when starch paste containing the dye was dried down in the cold was due to the removal of water and the resulting increase in concentration of the soluble dye in the starch phase. The reverse transfer in the early stage of steaming was then ascribed to the take-up of water from steam, predominantly by the starch, which has a much greater water absorption than has cellulose acetate. These factors give the incentive to transfer in a particular direction, but the transfer will be impeded when drying is carried so far that the dye in one phase or the other is unable to move. To illustrate this, after a layer of dye-containing paste had been spread on a sheet of cellulose acetate, drying was carried out under three different sets of conditions, and steaming followed in the usual way. There were two sets of experiments in which the drying took place at room temperature—one into an atmosphere of 75% R.H., so that complete drying required 40 hr., and one into an atmosphere of 65% R.H., which shortened the drying time to 18 hr. In a third set, with drying at 55°c. and 30% R.H., the operation was much more rapid (3·5 hr.).

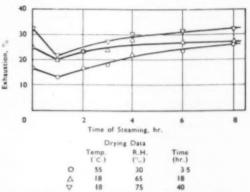


Fig. 14— Effect of Conditions of Freliminary Drying of Starch Paste upon Transfer of Pure Solacet Fast Crimson B to Cellulose Accetate during Steaming

It can be seen (Fig. 14) that the slowest drying operation, giving full opportunity for the dye to remain mobile in the water-swollen starch and cellulose acetate until diffusion is completed, gives rise to a higher initial exhaustion than do operations in which water is taken out of the system more quickly. What is more interesting is that the level of swelling seems to be set more or less irreversibly by the drying conditions, so that in the subsequent steaming operation further transfer of dye, first in one direction and then in the other, is greatest with material dried most slowly at room temperature and least with material dried most quickly at a higher temperature.

## EFFECT OF SWELLING AND SOLUBILISING ASSISTANTS

A small selection was made from the great variety of assistants in this field, and the effects on Solacet Fast Crimson B and Solacet Fast Scarlet B were investigated. Of these agents, Solution Salt SV (sodium NN-dibenzylsulphanilate) may be regarded as acting primarily as a dispersing agent for the dye, sodium thiocyanate as acting primarily as a swelling agent for the cellulose acetate, and diethylene glycol and ethyl lactate as combining the two functions in different proportions. It will be seen (Fig. 15-17) that sodium thiocyanate is alone effective in causing a really large transfer of soluble dye to the cellulose acetate as a result of steaming. In the experiments represented by Fig. 17 it was desired to avoid the complication that might come from prolonged drying in the cold of starch paste containing a powerful swelling agent in contact with the cellulose acetate. these experiments, therefore, the original method of preparing separate solid starch films and then

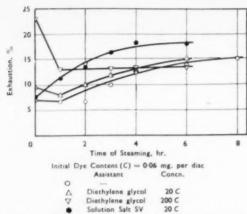


Fig. 15—Effect of Assistants on Transfer of Pure Solacet Fast Crimson B during Steaming

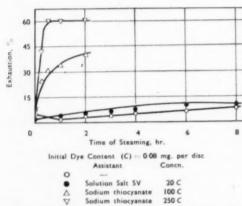


Fig. 16— Effect of Assistants on Transfer of Pure Solacet Fast Scarlet B during Steaming

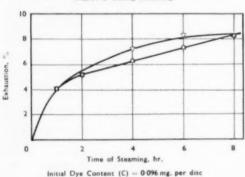


Fig. 17 — Effect of Assistants on Transfer of Pure Solacet Fast Scarlet B during Steaming. Solid Starch Film on Cellulose Acctate

attaching them to the cellulose acetate was employed. The other assistants applied by this method have comparatively little effect in the proportions used. With the drying-down method of preparation (Fig. 15) there are two interesting observations. Diethylene glycol, when present in

sufficient proportions, can cause a great increase in dye transfer on drying down, a correspondingly large reverse transfer when steaming begins, and the establishment, early in the steaming, of equilibrium transfer, the only well marked equilibrium that has hitherto been observed for the soluble dyes.

Solution Salt SV cuts out the reverse transfer of dye in the early part of the steaming. This may be because the dye is deposited in the exterior zone of the cellulose acetate in an especially finely dispersed form during drying, so that when steaming begins the dye can diffuse forward into the cellulose acetate as rapidly as it can back to the starch. It is obvious that there is scope for a long and detailed study of the effects of swelling and dispersing assistants.

## DESORPTION OF DISPERSE DYES FROM CELLULOSE ACETATE TO STARCH

Duranol Red 2B was chosen for this study. The pure dye was ground in water for 20 hr. The dye was introduced into the cellulose acetate in two ways—

(i) By first steaming the film of cellulose acetate in contact with a film of starch containing the dye (printing transfer)

(ii) By dissolving the dye along with the cellulose acetate in acetone before spreading the resultant solution (dope dyeing).

On the dye-containing film a layer of plain starch paste was spread, and this was dried down in air at room temperature. The compound film was then steamed for the desired time, and the exhaustion of the dye from the acetate to the starch was determined. It will be seen (Fig. 18) that, with

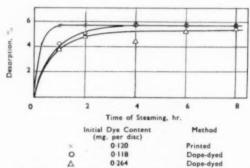


Fig. 18—Desorption of Pure Duranol Red 2B from Cellulose Acetate to Starch during Steaming

the same concentration of dye in the acetate layer at the beginning, the transfer to starch is distinctly more rapid from acetate which has been dyed by printing transfer than from acetate dyed in the dope. Nevertheless, the equilibrium distribution appears to be independent of the method by which the dye was first introduced into the cellulose acetate. The experiments in one series were extended, and the equilibrium partition of dye between the two phases, with different concentrations of dye in the system, is given by the isotherm in Fig. 10 in comparison with the isotherm which resulted from adsorption experiments. It will be



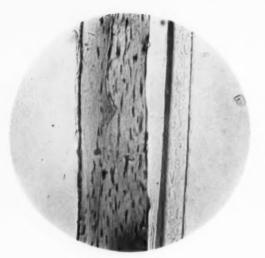


Fig. 19 Before Steaming

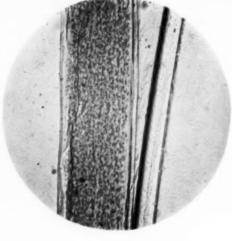


Fig. 20 After 1 hr.

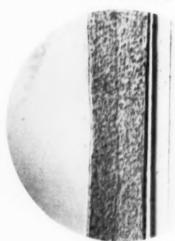
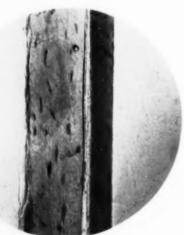


Fig. 21 After 2 hr.



Fig. 22 - After 4 hr



Ftg. 23 - After Shr.

Fig. 19-23 — Diffusion of Solacet Fast Scarlet B from Starch to Cellulose Acetate during Steaming. The broader, more irregular section is the starch film. Contiguous layers of starch and cellulose acetate have been separated slightly before photographing, so that the contact boundary of each phase should be better defined.

seen that there is a consistently higher proportion of total dye in the acetate phase when equilibrium has been reached by desorption.

#### EXAMINATION OF FILMS IN CROSS-SECTION

In these experiments, the starch-cellulose acetate system, containing dye, was prepared and steamed for the desired time in the usual way. On removal from the steam, the combined films were allowed to cool without separation, embedded in wax (90% paraffin (m.p. 60°c.) + 10° beeswax), cut in sections 60 u. thick perpendicular to the face, cleared, and mounted in Euparal (Flatters & Garnett). In mounting, it was generally found advantageous to separate the section of starch film from the contiguous section of acetate film for a short distance, so that superficial adsorption on the acetate film should not be masked by optical effects at the boundary arising out of the difference in refractive index between the two phases. One example each of disperse and of soluble dyes was studied.

#### Duranol Red 2B

The results here are straightforward and do not call for reproduction of the sections. On drying, but before steaming, there is a very faint coloration of the acetate film at the interface between the two layers. Dye has penetrated halfway through the acetate film in 15 min. steaming, has almost reached the opposite side in 60 min., and is uniformly distributed in the acetate, with a faint coloration in the starch, at the end of 4 hr. It was noticed that when drying-down had been completed, but before steaming, the disperse dye (ground for 20 hr.) present in the starch imparted to it a rather faint purplish-grey coloration. After being steamed for 15-30 min., the dye remaining in the starch had undergone further dispersion and now gave rise to a clear brilliant red, similar to the colour of the dye in the cellulose acetate. This dispersion by heat in the starch layer before transfer to the acetate was also found with other members of the disperse series.

#### Solacet Fast Scarlet B

The results with this dye are illustrated by the photomicrographs in Fig. 19-23. It will be seen that there is some penetration into the cellulose acetate after drying-down and before steaming. Steaming for 1 hr. causes little apparent movement of the dye and little deepening in the colour of the dyed portion of the acetate. Only after 2 hr. steaming has the dye penetrated further into the acetate film, and complete penetration through the acetate film requires 6-8 hr. If the total proportion of dye in the system is low, the return of the dye to the starch layer during the first hour of steaming is seen to be almost complete. Examination of cross-sections obtained from the films during the course of desorption experiments were not very informative, since the proportion of the dye which is transferred to the plain starch, either on drying-down or on steaming, is so small that it is only just visible in the microscope. There is little evidence, even with short times of steaming, of localised distribution of dye which

has migrated from the cellulose acetate into the starch. This dye appears to be liberated so slowly from the acetate, and to diffuse so readily in the starch, that the latter is uniformly coloured at any time during the steaming. With prolonged steaming there is no evidence of aggregation into visible particles of the dye which has thus migrated.

#### PARTITION OF DISPERSE AND SOLACET DYES BETWEEN AMYL ACETATE AND WATER

The analogy between the dyeing of cellulose acetate from aqueous solutions or suspensions and the partition of a common solute between water and a non-miscible solvent has been considered and investigated quantitatively by a number of workers 6, 14, 18. When the partition of Dispersol Fast Scarlet B (pure dye ground in water) between water and amyl acetate was determined in the usual manner, the whole of the dye originally introduced into the system was found dissolved in the ester phase, that remaining in the water being too small to be detected by the absorptiometer. With Solacet Fast Scarlet B, this situation was reversed (Table III).

TABLE III Partition of Solacet Fast Scarlet B between Amyl Acetate (50 ml.) and Water (150 ml.)

Total Dye in System (g.)	Dye in Amyl Acetate (g./50 ml.)	Dye in Water (g./150 ml.)
0.0103	0.0001	0.0100
0.0206	0.0003	0.0201
0.0309	0.0004	0.0290
0.0515	0.0007	0.0505
0.0618	0.0009	0.0601

Partition, in so far as it can be calculated reliably with such small concentrations of dye in the amyl acetate, is not quite independent of the weight of dye in the system. The fact that the sum of the weights of dye in each of the layers is slightly less than the total weight originally introduced into the system was accounted for by the visible concentration of undissolved dye at the interface between the two phases.

#### Discussion

BEHAVIOUR OF CELLULOSE ACETATE FILM ON STEAMING

In printing systems two features recur persistently and serve to emphasise the difference between dye transfer from thickener to fibre during steaming and dye transfer from bath to fibre during dyeing. If water-swollen thickener in the former type of system is analogous to the dyebath, then, ignoring for the time being any influence which may be exerted by the thickener molecules themselves on the dye, these distinctions are-

(i) The very low "liquor : goods ratio" in the printing system with the consequent high concentration of the "dyebath"

(ii) The changes in volume which occur during the steaming of a print, in both thickener and fibre phases, changes which have no exact counterpart in any dyeing procedure with the possible exception of pad-steam methods.

Item (i) has already received a good deal of comment in previous papers, but (ii) calls for further explanation. During the impression there is, in most thickener pastes, a high proportion of water. If the print is dried, water is lost, the dye in the paste becomes more concentrated, and this has been shown, in the present paper for soluble dves and by Patel and Turner 4 for acid dyes, to lead to some transfer of dye to the fibre substance. On introduction into saturated steam, thickener\* and fibre substance take up water to different extents, the dye becoming again capable of migration. Evidence obtained in this laboratory by G. D. Myers and R. E. Pomfret shows that the water content of films of regenerated cellulose and also of films of dried starch, examined apart, increases continuously over long periods of steaming. So far it has not been possible to determine the changes of water content in these two materials when they are maintained in contact in steam. It would probably be true to say that, in the absence of a deliquescent, the water content of regenerated cellulose in contact with steam will never reach the value which is attained when it has been immersed in liquid water at 100°c, for a few minutes. In a model dyeing system, the volume of the dyebath is substantially constant from beginning to end of the dyeing operation, and, after a short initial period, the volume of the fibre-substance changes only slightly. Allowances for this difference in behaviour will need ultimately to be made before dyeing and printing systems can be accurately compared.

In changing from regenerated cellulose to cellulose acetate as the fibre-substance, while still retaining starch as the thickener, a system is produced in which there is a much greater disparity between the water-absorbing capacity of the constituent phases. This is important in itself, but certain qualitative experiments have revealed an even more interesting state of affairs, and have, for the first time, shown how the water-uptake of one phase is affected by that of the other.

Delustring of cellulose acetate at temperatures approaching 100°c, takes place only if sufficient water is present in the material. If a dry film of the acetate is introduced alone into saturated steam, there is pronounced delustring in about 30 min., which proves that the acetate is able to take up sufficient water from the steam for this effect to be produced. If, however, the acetate film is steamed in contact with a film of starch, initially dry, delustring will not take place even when the steaming is continued for 13 hr., the starch thus acting continuously as a desiccant for the acetate and keeping its moisture content at a very low level. This evidence shows that it is not necessary for the cellulose acetate to be swollen even to the limits of its water-absorbing capacity in order to take up disperse dye, and may be pertinent when the "solvent" properties of cellulose acetate for this type of dye come to be discussed. It helps also to explain the low exhaustions of the soluble dyes, for these, being soluble in water, will tend to remain

in the phase where this water is present in the greatest proportion.

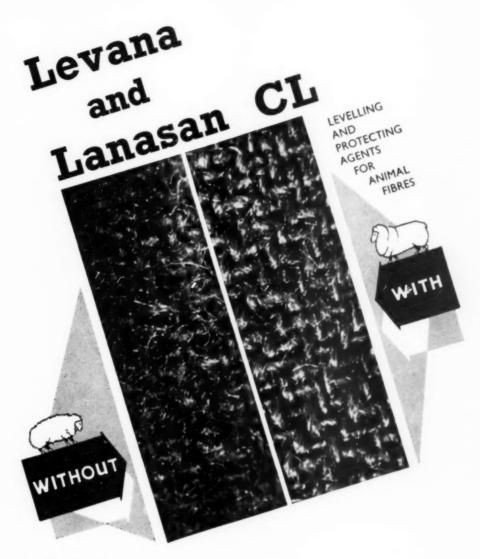
#### EFFECT OF GRINDING

The effect on the course of dyeing of the degree of subdivision of sparingly soluble azo and anthraquinonoid dyes used for cellulose acetate has been studied by Clayton <sup>10</sup>, Kartaschoff and Farine <sup>14</sup>, and Vickerstaff and Waters <sup>18</sup>. Coarse and mechanically unstable suspensions make accurate dispensing difficult and increase the risk of unlevel dyeing: increasing fineness generally brings about increasing rate of transfer of dye to the fibre and, in consequence, an increase in the exhaustion for fixed dyeing times short of equilibrium.

Clayton 10 has shown that  $\beta$ -aminoanthraquinone when applied from fine aqueous suspensions gives much paler shades than when applied from true solution in alcohol. According to Kartaschoff and Farine 14, the suspended particles of dye tend to collect at the surface of the fibre and then to disappear when the temperature is raised, imparting to the fibre-substance the same intense colour that they impart to liquids which will dissolve them. Vickerstaff and Waters 18 have found little evidence of this preliminary collection of dye at the fibre surface. Kartaschoff is inclined to the view that the influence of fineness on the rate of dyeing is of secondary importance, while Vickerstaff and Waters have shown that the longer the time taken for grinding in the presence of a dispersing agent (Lissapol LS, ICI), the more rapid the dyeing even after dyeing for 24 hr., and suggest that the dye is present in a polydisperse system, the smallest dye particles being accepted by the fibre. As these are removed from the bath, further dispersion of the larger particles is induced. The more complete the preliminary mechanical subdivision, the more rapidly can the very fine "primary" particles be furnished to replace those taken out of the bath by

Results in the present work, on film-to-film migration of the dye, tend to agree more closely with those of Vickerstaff and Waters than with those of Kartaschoff, although there are important differences. Thus, when 1-anilino-4-methylaminoanthraquinone is applied from aqueous suspension at 85°c., the equilibrium value for exhaustion increases with increased time of grinding. With 1-amino-4-hydroxyanthraquinone (pure Duranol Red 2B), ground in water in the absence of a dispersing agent and applied from a starch film by steaming, the equilibrium distribution between starch and cellulose acetate is independent of the time of grinding. On the other hand, the time within which the equilibrium is attained diminishes as the time of the preliminary grinding is increased up to 20 hr. Further grinding has no effect on the dyeing characteristics of the suspension, and it is probable either that little further comminution can be effected, or that the polydisperse system has reached equilibrium in 20 hr. In the dispersions of Vickerstaff and Waters there were considerable proportions of primary particles, capable of dyeing. or they could be readily furnished by a shift in the equilibrium, since dialysis through Cellophane removed up to 50% of the dye. In the printing

This excludes, of course, printing styles with non-aqueous media, or styles in which the thickener, such as albumin, methyl cellulose, etc., may be insolubilised by heat.



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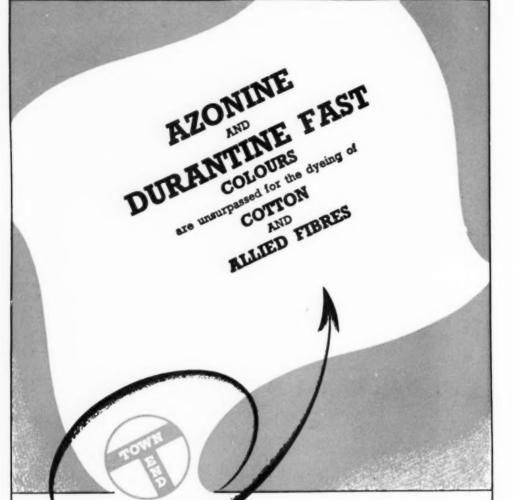




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experiments, there is evidence of the active dispersion of the dye before it leaves the starch film during steaming, for the colour of the dye in this film turns from a faint blue-grey to an intense red within half an hour after steaming has begun. The exhaustion of dye from coarse suspensions is different in the experiments of Vickerstaff and Waters 18 from that in the present experiments. Thus, unground 1-anilino-4-methylaminoanthraquinone suspended in water in the presence of Lissapol LS and applied at 85°c. gives negligible exhaustion in 30 min. and only 2% in 24 hr. When applied from starch film by steaming, the exhaustion of crystallised Duranol Red 2B ground for 5 min. in water alone is 20% for 30 min. and 75% for 8 hr. steaming. The greater transfer of dye by the printing procedure may be explained by the higher temperature of application, a specific dispersing action of the starch, the high concentration of dve due to the small volume of the starch layer, and the fact that dye particles small enough to be taken up by the acetate have only a short distance to diffuse in order to arrive at the surface of the fibre substance.

#### EXHAUSTION VALUES FOR DISPERSE DYES

The high values for equilibrium exhaustion in the present work agree in general with the observations of other workers who have studied the dyeing of cellulose acetate with disperse dyes. Representative values are 55–83% for various Celliton and Celliton Fast (IG) dyes <sup>16</sup>, up to 96% for 1-anilino-4-methylaminoanthraquinone <sup>18</sup>, and 70–95% for the commercial forms of a number of Duranol and Dispersol dyes <sup>28</sup>. Where comparisons can be made, they indicate a closer agreement between the results of experimental dyeing applications and application in the present experimental printing system than has been found for direct <sup>1</sup>, acid <sup>4</sup>, and Indigosol <sup>3</sup> dyes (Table IV).

TABLE IV

	Equilibrium Exhaustion (%)			
Dye	Dyeing at 85°c. (commercial brands) <sup>28</sup>	From Starch by Steaming (pure dye)		
Duranol Red 2B 300*	83.5	90		
Dispersol Fast Scarlet B 150*	96	87		
Disperse Dye A		87		

<sup>·</sup> Brand strengths refer to dyeing experiments only

In spite of the physical differences between normal cellulose acetate rayon and the films used in the present experiments, the saturation value for disperse dyes is not greatly changed. Vickerstaff has given this value as 10.8% of commercial Duranol Red 2B 300 for the acetate rayon used in his dyeing experiments. When this brand is analysed there is found to be 15% of pure dye present, so that the saturation value in terms of pure dye is 1.62%. In the present work, the corresponding value is 1.59%.

#### RATE OF DYE TRANSFER

Comparison of published values for the rates of dyeing of selected dyes with the rate of dye migration on steaming in the present system are difficult, because the dimensions and other properties of the two kinds of system differ so much. Nevertheless, Gill and Peters <sup>28</sup> found that the ratio of the times of half-dyeing of Dispersol Fast Scarlet B and Duranol Red 2B was 1:1-6. Times of half-transfer in the present system during steaming (for identical preliminary grinding times and for the same initial dye concentration in the starch film) were also in the ratio 1:1-6.

Earlier observations of Green and Saunders <sup>30</sup> and of Ellis <sup>31</sup> had led to the general opinion that disperse dyes are taken up relatively more slowly as their molecular weight increases. This is supported in the comparison of Dispersol Fast Scarlet B with its higher homologue, Disperse Dye A, where a change in the substituent group from  $C_2H_5$  to  $C_6H_{13}$  greatly diminishes the rate of transfer during steaming. Comparisons of this kind are of limited validity only: this is demonstrated by the results of Vickerstaff and Waters <sup>18</sup> for some derivatives of 1-amino-4-anilino-anthraquinone.

#### TRANSFER MECHANISM OF DISPERSE DYES

Alternative possibilities for the dyeing mechanism with disperse dyes on cellulose acetate yarn have been reviewed by Vickerstaff and Waters <sup>18</sup>. The most probable appear to be (a) solid solution and (b) adsorption of dye molecules on specific sites within the fibre, leading to equilibrium conditions best defined by the Langmuir isotherm. There is experimental evidence to support each alternative. In the absence of molecular aggregation in either phase, the process of solid solution requires the following conditions to be fulfilled at equilibrium—

Conen. of dye in fibre Solubility of dye in fibre Conen. of dye in bath Solubility of dye in bath

so that (i) equilibrium exhaustion of the system is independent of the total amount of dye present and (ii) the point at which saturation of the fibre with dye occurs will be sharply defined. An experimental isotherm which fulfils these requirements is given in Fig. 8. The Langmuir isotherm resembles the solid solution isotherm over part of the concentration range, but the inflexion corresponding to saturation is not abrupt. The experimental results in Fig. 8 appear, therefore, to exclude mechanisms other than solid solution. Previous evidence of constant partition of a number of organic solutes between cellulose acetate and water or alcohol must sometimes be criticised because (a) low working temperatures make the establishment of equilibrium slow and uncertain, (b) measurements have been made over too restricted a concentration range, and (c) some of the solutes may cause swelling of the cellulose acetate.

The thorough examination of typical disperse dye systems <sup>18</sup> led to conflicting evidence as the experimental conditions were changed, some results suggesting a process of solid solution, others Langmuir adsorption. It was suggested that the true partition behaviour was being obscured by the collection of undissolved dye particles at the surface of the fibre.

Vickerstaff has objected to the unqualified acceptance of the solid solution theory, principally because— (a) if the dye is readily soluble in the acetate phase, it is so sparingly soluble in water that the final exhaustion should be nearer 1000, than is actually found; (b) constant partition may be simulated because some of the dye particles in the bath remain too large ever to pass into the cellulose acetate. These objections should apply also to dye transfer in the present printing systems. This difficulty probably comes from the attempt to find an unjustifiably close analogy between a dyeing or a printing system on the one hand and a system such as disperse dye in a water-amyl acetate mixture on the other. In the dveing and printing systems, the aqueous phase does not consist of water and dye alone. In the experimental systems of Vickerstaff and Waters there is also present a dispersing agent; in the printing systems there is the thickening agent. Either of these additions seems to have the effect of causing an increased solubility of the disperse dye in the aqueous phase. Thus, when Vickerstaff and Waters employed solutions of Lissapol C to desorb a disperse dye from dyed cellulose acetate, appreciable proportions of the dye were removed, whereas Burns and Wood 15 found that plain water caused, no removal. In the present work, it is seen that, when dyed cellulose acetate film is steamed in contact with a plain starch film, a small but significant proportion of the dye passes into the

The printing system used in the present work may appear to be so much more complex than a simple system of two immiscible solvents that the very simple dye-partition behaviour observed experimentally may be unexpected. It should be noted that—(i) at equilibrium, the concentration of dye in the starch phase is low, so that all the molecules may finally be present unassociated; (ii) volatile dyes have been used, and there is evidence that these are highly dispersed during steaming (p. 249); and (iii) the temperature is well above that which can normally be employed for the dyeing of cellulose acetate.

A simple experiment indicates that there are unlikely to be aggregates of dye molecules in the starch which are incapable of being dissociated into single molecules, and therefore incapable of diffusion from starch to cellulose acetate, during steaming. A steaming experiment was carried out until equilibrium distribution of the dye had been established. The starch layer, containing a small residue of the dye, was then removed and attached to a fresh piece of plain cellulose acetate film, and the two were steamed together. A further quantity of dye left the starch and entered the cellulose acetate.

As will be described in a later paper, constant partition of the same disperse dyes is observed on steaming corresponding starch-nylon systems.

TRANSFER BEHAVIOUR WITH SOLUBLE DYES

In comparison with the disperse dyes, the corresponding soluble forms when applied without the addition of electrolyte have been seen to give very low transfers to the cellulose acetate from the starch. The maximum exhaustion attained at the end of some 8 hr. steaming may be well below 10%. As in dveing, the overall exhaustion can be increased markedly by additions of sodium chloride in the thickener phase, though in general the characteristic shape of the transfer curve is not greatly modified. In none of the present experiments, either with salt or without, can a true equilibrium condition be said to have been reached. Although these general results compare well with the behaviour recorded by Marshall 20, Fowler and Michie 21, and Abbot 32, the low rate of transfer and the low level of exhaustion are particularly striking in the present printing experiments, where the "bath" is so small in volume and therefore so concentrated both with respect to dye itself and also with respect to salt when this is present.

It is obvious that the single sodium sulphuric ester group which has increased the solubility of the essential dye substance in water completely inverts the distribution properties, as it does in the amyl acetate-water system. This change in behaviour when the water-solubility of the dye is increased is another confirmation of observations which have been made ever since the dyeing behaviour of cellulose acetate has been studied. The soluble dyes themselves are not, in general, freely soluble in water, especially in the cold. The dyeing isotherms for these dyes on cellulose acetate show many formal similarities with the corresponding isotherms for direct cotton dves on cellulose 21. Affinities are difficult to determine for all except weak dyebaths, since aggregation of the dye takes place in stronger baths. In dyeing without salt the effect of the relatively high surface potential of cellulose acetate against water needs to be taken into account, but this is likely to be low when dye transfer is taking place in a printing system and, in addition, the ions are univalent. It was unfortunate that the influence of boundary potential on dye transfer could not be demonstrated qualitatively, as in experiments with the direct dyes, by the use of sodium alginate as the thickener, for this substance decolorised both disperse and soluble forms with unexpected rapidity and completeness.

With the dye mobile in water, the drying-down of the starch paste containing the soluble dye in contact with the cellulose acetate before steaming results in a considerable transfer of dve to the fibre-substance in the cold. Under the microscope this dye is seen to be deposited in a very thin superficial layer of the cellulose acetate, a layer which does not increase in thickness until the temporary reversal of migration at the beginning of steaming has been completed. From this it would seem that, in the air-dry double film, both the starch and the cellulose acetate layers initially contain insufficient water for any movement of the dye. On steaming, the starch swells first, and increases markedly in volume, and since the acetate also begins to take up water, the dye in it is released and desorbs to some extent into the starch. It should be noted that, apart from the different constitution and properties of the fibresubstance, the present procedure for making the

experimental system would be expected to give rather different results from those given by the system which has been most commonly used in earlier investigations, when cellulose was the fibresubstance. Here the cellulose was in the form of Cellophane, which was used directly in the water-soaked condition, and on which a solid film of thickener was pressed. In the earlier experiments, therefore, the fibre-substance is fully swollen from the beginning, and dve can enter or leave it with much greater freedom during the early stages of the steaming. This has been shown by some recent work of Mr. H. B. Hallows in this laboratory. He has followed the movement of a direct cotton dye between starch and cellulose by observing the system in cross-section for (a) a solid starch film containing dye in contact with fully soaked Cellophane, and (b) a system prepared by spreading the wet paste on Cellophane and drying in the air at room temperature. With (a) dye began to migrate into the Cellophane as soon as steaming commenced, and spread steadily into the thickness of the cellulose film as steaming continued. With (b) some dye was transferred to the cellulose during the drying in the cold. It resided in a thin layer behind the starch-cellulose boundary. On steaming, this layer did not increase in depth for more than an hour, and even after 8 hr. the dye had not progressed so far into the cellulose layer as it had in (a). This forms another part of the evidence that has been accumulating to show that, unless deliquescents are present, the water content and degree of swelling of a fibre-substance in steam will never be so great as in an aqueous dyebath.

Because of these complications, and because of the fact that it was impossible to attain equilibrium in the system before marked deterioration of the starch had set in, it was not possible to express the fundamental dyeing properties at equilibrium by means of an isotherm. With Soluble Dve A (Fig. 7 and 13), it might be expected that the effect of the larger hydrocarbon substituent, as compared with the lower homologue, Solacet Fast Scarlet B, would be to increase the exhaustion and also the salt sensitivity, in so far as salt may be expected to increase the degree of aggregation in the starch film. This appears to be confirmed by the results of the transfer experiments, but it is also found that the solubility of the higher homologue in water, both cold and at 95°c., is greater than that

of the lower.

EFFECT OF SWELLING AND SOLUBILISING AGENTS

This subject has been only lightly touched upon in the present account. Special techniques will probably need to be devised for a more thorough study, because some of the auxiliary substances cause great modification in the properties of the thickener films, so that comparison with the more conventional experiments may be very difficult. A much fuller investigation is required to decide how far the effect of one auxiliary substance may be compared with that of another.

The effects of a small selection of these agents have been measured for the two dyes Solacet Fast Crimson B and Solacet Fast Scarlet B. Solution Salt SV is the common addition for each dye, and it is seen to be specifically less active with the second With both dyes, and especially with Solacet Fast Crimson B, it has already been seen that the addition of the Solution Salt SV causes little change in the exhaustion during drying of the paste in the cold, but suppresses the reverse migration of dye during the early stages of steaming, and increases the rising exhaustion as steaming is prolonged. This can best be explained by assuming that the dispersing action of the Solution Salt SV on the dye particles is not very pronounced at low temperatures. Then the dye, which has been transferred to the outer layer of the cellulose acetate more or less normally during the drying, is at once capable of diffusing further into the acetate when steaming begins. A delay in the forward migration, which would be expected if the larger dye particles had to wait for dispersion by heat alone, or for the acetate to take up water, is

On the other hand, the swelling action of sodium thiocyanate upon the cellulose acetate, once sufficient water is obtainable from the steam, is reflected by an unusually rapid and large migration of dve to the fibre-substance. Diethylene glycol seems particularly to affect the earlier stages of dye transfer (Fig. 15). Thus Solacet Fast Crimson B is encouraged to migrate into the cellulose acetate in considerable proportions during the drying process, to return readily to the starch in the first hour of steaming, and then to remain at a steady concentration in the acetate if steaming is continued. It seems that this solvent can greatly encourage migration in either direction while it is present. It is, however, the only volatile auxiliary among those already mentioned, and after exerting its early effect, it has probably evaporated. In experiments where the solid starch film is pressed on to the cellulose acetate, and there is little opportunity of swelling before steaming is begun, the diethylene glycol and the other solvent, ethyl lactate, are seen to have little

In addition to the acknowledgments already made, we express our gratitude to the Foreign Education Committee of Bombay and the City of Manchester Education Committee for grants which have enabled one of us (E.H.D.) to take part in this work.

DEPARTMENT OF TEXTILE CHEMISTRY COLLEGE OF TECHNOLOGY University of Manchester

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#### Notes

#### Proceedings of the Council

At a meeting of the Council held at the offices of the Society, 19 Piccadilly, Bradford 1, on 10th June 1953, the proceedings included the following items

Mr. G. E. Holden-Council stood as a mark of respect to the memory of Mr. G. E. Holden.

WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL- The resignation of Dr. H. H. Hodgson was accepted with regret, and Mr. G. B. Angus was nominated to serve on the Committee.

Membership-Six applications for ordinary and one for junior membership were approved.

#### Meetings of Council and Committees Iune

Council-10th

Finance-23rd Publications-23rd

Colour Index Editorial Panel - 5th

Mercer Lecture-12th

Review of Textile Progress-18th

Textile Printing Symposium— 19th

Terms and Definitions-30th

Joint Conference with Textile Institute-25th

#### Deaths

We regret to report the loss by death of Messrs. R. W. Burnett, E. Turnbull, and A. G. Winning.

### John Garnett Hopkinson

Honorary Secretary of the Society Mr. J. G. Hopkinson, B.A., A.R.I.C., has accepted Council's invitation to serve as Honorary Secretary of the Society in succession to Mr. J. Barritt, B.Sc., A.R.C.S., A.R.I.C.

The resignation of Mr. Barritt has been accepted by Council with very great regret, but Council has appreciated that it is unavoidable, owing to Mr. Barritt's additional responsibilities arising out of his recent appointment as Assistant Director of Research at the Wool Industries Research Association and to his commitments as a Part I editor of

the Second Edition of the Colour Index

Mr. J. G. Hopkinson is the elder son of Mr. G. G. Hopkinson, a former Vice-president of the Society and himself at one time Honorary Secretary. After over three years' service in the Royal Navy, Mr. J. G. Hopkinson graduated at Oxford with Honours in Chemistry, and later spent some time in studying dyeing and textiles at Leeds University and Bradford Technical College. He is a director of Messrs. Hopkinson & Shore Ltd., dyers and finishers of Brighouse, Yorkshire.

#### John Barritt

#### Chairman of the Publications Committee

Mr. J. Barritt has been elected Chairman by the Publications Committee in succession to Mr. C. L. Bird. Members of the Society will share Council's warm gratitude to Mr. Bird for his services in guiding the destinies of the Society's Journal, after having edited it for geveral years, and will be especially pleased to learn that Mr. Barritt is thus maintaining an active interest in the affairs of the Society.

#### Symposium on Textile Printing

A notice and application form for this symposium, to be held at the Hotel Majestic, St. Anne's-on-Sea, from Wednesday to Friday, 23rd-25th September 1953, was enclosed with the June issue of the Journal. Further copies may be obtained from, and it is requested that completed application forms should be returned as soon as possible to, the Society of Dyers and Colourists, 19 Piccadilly, Bradford I, Yorkshire.

#### Joint Conference Committee

The following members have accepted Council's invitation to serve on the organising committee for the Joint Conference to be held by the Textile Institute and the Society in 1954-

Mr. F. Crompton Mr. G. E. Styan Mr. F. Farrington Mr. A. G. Tyler Mr. C. F. Ward Dr. C. S. Whewell Mr. N. Hamer Mr. J. W. Reidy Dr. C. B. Stevens Mr. G. S. J. White

The representatives of the Textile Institute on the Joint Conference Committee are-

Mr. J. Boulton Mr. W. Crossley Mr. C. Howden Mr. A. W. Bayes Mr. F. Millard Mr. J. H. Black Mr. J. Boyd Mr. L. Morris Mr. V. Castle Mr. B. H. Wilsdon

At the first meeting of the Committee, held on 25th June 1953, Mr. J. Boulton was elected Chairman.

### Reports of Sections for the Session 1952-1953

#### HUDDERSFIELD SECTION

During the past session we have had nine lectures, and, as in previous years, we have had NOTES 255

joint meetings with two other Societies. The first was held jointly with the Huddersfield Textile Society, when Dr. C. S. Whewell gave us a very interesting talk on *The Future of Textile Finishing*. On the occasion of our joint lecture with the Huddersfield Section of the Royal Institute of Chemistry, we were favoured by a visit from Sir Wallace Akers, F.R.S., who gave a "follow-on" lecture to the one he gave us two years ago.

The standard of lectures has been high, and the Committee has again tried to arrange lectures to appeal to the practical man. In December, Professor Speakman gave us a very interesting talk, revealing some of the lines on which he and the wool growers were working so as to produce the types of fibre that were required in this country.

As in previous years, we held our local dinner in November. This was very well supported, and we were favoured by the presence of the President of

the Society.

In conclusion, I should like to thank all the officers and members of the Committee for their valuable assistance, as without their help, it would not have been possible to report another successful session.

H. Turner Honorary Secretary Huddersfield Section

LONDON SECTION

The London Section is glad to report that the 1952–53 session has been in many ways an outstanding one. Firstly, both the Mercer Lecture and the Society's Annual Meeting and Dinner were held this year in London. Secondly, the new series of "London Lectures" was inaugurated, the first lecture being delivered on 10th March 1953 by Sir Ernest Goodale. We are happy to report that this lecture was a complete success; over 300 persons were present, and there is no doubt that both the lecturer and his subject were greatly appreciated. We confidently look forward to the continuance of this series of lectures, and feel certain that they will enhance the prestige of our Society in this district.

The remaining lecture meetings of the Section have been held, as formerly, in the rooms of The Royal Society, and we should once again like to place on record our appreciation of the hospitality of this renowned body, and of the privilege of using their beautiful hall. The average attendance at these meetings—excluding the London Lecture—was 45 persons, and in this connection we should mention that this number represents 28% of those members, affiliated to our Section, who live within a reasonable distance of London. We feel that this attendance shows an encouraging sign of keen support by our members.

A lecture was given in Luton, as in previous years. This was reasonably well attended and we feel sure that members of the dye-using industries in that district appreciate our activities there.

Our social events have consisted, as in past years, of a dinner and dance early in the New Year, and a dinner for members only, following the Annual General Meeting of the Section. Both these functions were well attended.

We look forward to a successful season in 1953-54, when, in addition to the London Lecture, we hope to devote one of our meetings to a subject which will be of special interest to ladies.

H. W. Ellis
Honorary Secretary
London Section

#### MANCHESTER SECTION

Ten meetings have been held in Manchester this session, one jointly with the Lancashire Section of the Textile Institute and one in collaboration with the British Association of Managers of Textile Works. The external meeting at Leek was held jointly with the Leek Textile Society. The evening symposium held last March was so successful that a similar function was organised this session, on Radioactive Isotopes in Textile Technology. As a new venture, a more ambitious one-day symposium on Synthetic Fibres was held in the College of Technology, Manchester, on 13th March 1953. It was attended by approx. 260 persons and proved a great success. The reception given to these meetings, and to the Ladies' Evening (at which three papers on the miscellaneous applications of dyes and pigments were presented under the title "Penny Plain, Twopence Coloured"), indicates the growing popularity of meetings at which a given topic can be discussed from several points of view simul-

The Junior Branch meetings have proved very successful, although it is disappointing once again to record that the audiences consist largely of members more senior than those for whom the Junior programme is devised. Members are asked to do all they can to encourage potential Junior members to join the Society and attend meetings.

Our thanks are due to all those who have contributed to these meetings. As the North-Western Gas Board Showrooms are no longer available to us, our meetings are now held at the headquarters of the Textile Institute, to whom we express our gratitude for permission to use their very comfortable lecture theatre. We also thank Mr. J. M. Preston and the Manchester College of Technology for lecture and committee meeting facilities.

The Knecht Memorial prize was won for 1951–52 by Miss Margaret Middleton, who was a student at the Manchester College of Technology.

The Annual Dance was again held at the Grand Hotel, Manchester, on 26th February 1953. The occasion was thoroughly enjoyed by nearly 200 members and their guests.

Mr. F. Farrington, Chairman of the Section for the past two years, retired at the Annual General Meeting in April, and was succeeded by Mr. G. S. J. White, with Mr. J. Boulton as Vice-chairman. Mr. Farrington has conducted meetings with great efficiency and zeal, and the Section is indebted to him for the great interest he has shown in its affairs. For the final year of office of the present Honorary Secretary, Mr. H. R. Hadfield has been elected Honorary Assistant Secretary.

We congratulate Mr. F. L. Goodall on his election as President of the Society.

Since 18th April 1952, when there were 596 members and 27 Junior members, the membership of the Section has undergone the following changes up to 17th April 1953—

	-			
New members	***	***	***	+25
Transfers from o	other se	ctions	***	+ 5
Resignations	* * *	***	+++	-15
Transfers to oth	er secti	ons		-12
Deaths	***	***	***	- 4
Net decrease	***	***	***	- 1

The Junior Membership has increased to 28.

We regret to record the deaths of Mr. C. W. Bancroft, Mr. O. Gunnell, Mr. L. Frank, and Mr. L. A. Lantz.

A. S. Fern

Honorary Secretary Manchester Section

#### MIDLANDS SECTION

Another successful session is reported. Nine lectures in all have been given, in Leicester (2), Loughborough (2), Nottingham (2), Derby, Hinckley, and Kidderminster. The last two were held in conjunction with the local section of the Textile Institute, and that at Derby with the British Association of Chemists. It is, however, regretted that an increase in the proportion of practical papers did not bring the extra response that was anticipated. Indeed, the average attendance fell slightly, which was disappointing in view of the high level of the papers.

On the social side, the Dinner Dance in November and the Dinner in March were highly successful.

Little change is to be reported in the total membership of the Section, new members being largely offset by transfer to other sections and by resignations. We also regret to report the loss by death of Mr. F. Hern.

New members	***	***	***	14
From other sect	ions	***	***	5
				19
Resignations	***	***		8
Death	***	***	***	1
Transfers to oth	er secti	ons	488	8
				17
Net increase		***		2

At the commencement of the session, our Section was delighted to be host to the Summer School of the Society at Loughborough College. A full report from the committee has already been published in the Journal. It but remains to add that local members who gave their time and talents to ensure its successful conclusion were much encouraged by the practical help given by members of other sections, and were well rewarded by the new friendships cemented with many of those taking the course.

Acknowledgment must also be made of the services of Messrs. W. A. Edwards, H. C. Olpin, W. Penn, and A. G. Tyler on various standing committees of the Society.

Mr. A. P. Kershaw has now relinquished the Chairmanship of the Section after two encouraging sessions, and our thanks go to him for his intense interest and enthusiasm in our work. In company with Messrs. Edwards and Olpin, he has been a regular attender at Council, and we are happy to record his election as an Ordinary Member of that body announced at the last Annual General Meeting of the Society. He is succeeded as Chairman by Mr. Sydney R. Meadows.

At the Annual General Meeting of the Section, Messrs. A. P. Kershaw, A. S. Cluley, D. B. McPherson, H. C. Olpin, and A. G. Tyler retired from the Committee by rota. The last four named were re-elected together with Mr. M. Nutton. The latter fills the vacancy created by the elevation of Mr. Kershaw to ex-officio status.

Since this meeting we have received news that Mr. G. L. Thornton is shortly to leave the district. He has been with the Section for 24 years, during 16 of which he has served on the Section Committee. His uncompromising efforts on behalf of the Section will be greatly missed.

A. W. CARPENTER Honorary Secretary Midlands Section

<sup>1</sup> J.S.D.C., **68**, 513 (Dec. 1952)

#### NORTHERN IRELAND SECTION

The most pleasing feature of the 1952–53 session has been increased attendance at lectures. This, we believe, has been due to the members' appreciation of our efforts in providing for them lectures with a very practical flavour.

It is also pleasing to report that, although we lost a few members owing to the increased subscription, we enrolled quite a number of new members, with the result that we had an overall increase in membership of 19.

The Annual General Meeting and Dinner of the Section were held on 28th March 1953, when Mr. J. Montgomery was elected Chairman in succession to Mr. D. A. Derrett-Smith, who had graced this office for two years.

We had as our chief guests to dinner the President and the Honorary Secretary of the Society (Messrs. H. H. Bowen and J. Barritt) and Colonel A. O'N. C. Chichester, a prominent local industrialist. It gave us much pleasure to entertain them and other guests.

I would take this opportunity to express my thanks to the Chairman and members of committee for their sterling support and co-operation during the session.

James Porter Honorary Secretary Northern Ireland Section

#### WEST RIDING SECTION

Under the Chairmanship of Mr. S. L. Peel the Section has had a successful, active year.

Ten lecture meetings have been held, including a lecture on the evening of the Annual General Meeting, when Mr. C. O. Clark talked on and exhibited *Historical Records* of interest to the Society. Eight lectures were held in Bradford, with an average attendance of 77. Two lectures held at Leeds University showed attendances of 43 and 68 respectively.

Other activities of the Section have included the social functions—the Supper and Smoker, which opened the Session, and the Ladies' Evening—both events being well supported by Members.

One experiment this year was the holding of a film evening, this replacing the usual joint lecture meeting between the Section and the Yorkshire Section of the Textile Institute. Two films were shown and a talk was given, followed by a buffet supper. This proved very successful, 254 members of the Sections and their ladies attending

This year has also seen the inauguration of an annual prize scheme for Junior Members of the West Riding Section as a whole, the prize money of ten guineas being donated by the Dyers and Finishers Association. On the evening of the presentation of the competition papers relating to the prize scheme, Council Members and Members of the Dyers and Finishers Association were present, and expressed great satisfaction at the quality of the papers submitted. They said they were pleased with the results of their efforts to encourage Junior Members.

The Section Committee met on five occasions.

G. E. STYAN

Honorary Secretary West Riding Section

**Tentative Definitions** 

The Terms and Definitions Committee will welcome comments on the following tentative definitions-

> 3 HUE (Revised)

That attribute of colour whereby it is recognised as being predominantly red, green, blue, yellow, etc.

27. PADDING

Impregnation of a fabric by passage through a liquid and subsequently or simultaneously through squeeze rollers.

28. TOPPING

The application of further colouring matter, not necessarily of the same hue or class, to a dyed substrate in order to adjust the latter to the desired final colour.

29. BOTTOMING

Dyeing a substrate for subsequent topping.

Note-In cotton bleaching "bottoming" is used to describe the preparation of the fabric for bleaching, a "well bottomed piece' being one which is thoroughly cleansed.

30. PACK DYEING

A method of dyeing in which the liquor is circulated through the goods.

Note-The use of the term "pressure dyeing" is deprecated in this connection.

31. PRESSURE DYEING

Dyeing under superatmospheric pressure, primarily with the object of raising the temperature of the dys liquor above its normal boiling point.

32. STAINING

(a) (in textiles) An undesirable local discoloration.

(b) Coloration of leather, paper, wood, and some other substrates by surface application of a dye solution.

33. AZOIC DYEING

The production of an insoluble azo compound on a substrate by interaction of a diazotised amine (azoic diazo component) and a coupling component (azoic coupling component).

34. AZOIC COMPOSITION

A composition containing the stabilised ingredients needed to form an azoic compound on a substrate.

A General Discussion on Dyeing and Tanning Leeds, 8-10th September 1953

The Faraday Society will hold this General Discussion in the Chemistry Department, Leeds University, from 2.15 p.m. on the Tuesday (September 8th) until 12.30 p.m. on the Thursday (September 10th). Application forms, obtainable from the Faraday Society, 6 Gray's Inn Square, London W.C.1, should be completed and returned as soon as possible. Preprints will be circulated before the meeting. Papers of special interest to members of the Society of Dyers and Colourists include the following-

include the Iollowing—

The Rate of Acid Absorption on Wood Fibrea

R. F. Hudson
The Combination of Acids and Dye Acids with Keratin

G. H. Lister and L. Peters

A Contribution to the Theory of Diffusion of Sorbed Substances into and
out of Fibrea

B. Olofsson
The Self-diffusion of a Dye Acid in a Polar Polymer Membrane
M. L. Wright

The Absorption of Sodium Sulphate and Sulphuric Acid by Hair
The Kinetics of Absorption of Water and Agneous Solutes by Dry Viscous
Cellulose
H. B. Mann and T. H. Morton
Theoretical Aspects of the Dysing of Cellulose Acetate Rayon
C. L. Bird, F. Manchester, and P. Harris
Researches in Monolayers. IV — A Study of Dyseing Processes by the
use of the Unimolecular Film Balance
M. M. Allingham, C. H. Giles, and E. L. Neustädter
The Absorption of Dyca by Crystala
A. N. Derbyshire and W. J. Marshall
The Solubility and Acticity of Orange II in Sodium Choica and Sodium
Sulphate Solutions
A. B. Meggy
The Selective Absorption of Optical Antipodes by Wool

The Solubility and Activity of Orange 11 in Somions.

A. B. Meggy
The Selective Absorption of Optical Antipodes by Wool
The Selective Absorption of Optical Antipodes by Wool
The Selective Absorption of Optical Antipodes by Wool
The Mucoid Material in Hides and Skins and its Significance in Tanning and Dueing
D. Burton and R. Reed
Mechanism of Absorption of Non-ionic Dyes by Polyethylene Terephthalate
M. J. Schuler and W. R. Remington
The Dyeing of Polyacrylonitrie Fibres with Anionic Dyes
R. H. Blaker, S. M. Katz, J. F. Lauchs, W. R. Remington, and H. E. Schroeder

The Dyeing of Synthetic Polypeptides
C. H. Bamford, J. Boulton, W. E. Hanby, and J. S. Ward

International Congress in Lucerne 17-19th September 1953

The complete programme of this congress on textile chemistry, dyeing, etc. is now obtainable from the Schweizerischer Verein der Chemiker-Coloristen (S.V.C.C.), Basle, Switzerland. Application forms, including hotel reservations, should be returned to the S.V.C.C. by 31st August 1953. Members of the Society of Dyers and Colourists will be warmly welcomed at this function, held under the auspices of the International Federation of Associations of Textile Chemists and Colourists, the S.V.C.C., and l'Association des Chimistes de l'Industrie Textile.

Third Continental European Fastness Meeting

The third meeting of E.C.E. (cf. J.S.D.C., 68, 306 (Aug. 1952)) was held in Bad Kissingen on 28-29th April 1953, the Deutsche Echtheitskommission being responsible for the arrangements. It was attended by fifty-five delegates from Belgium, Denmark, France, Germany, Holland, Italy, Spain, and Switzerland, as well as two observers each from Austria and Great Britain (Dr. P. W. Cunliffe and Mr. K. McLaren, Chairman and Honorary Secretary respectively of the Fastness Tests Coordinating Committee). results of the New York meetings of the Colour Fastness Subcommittee ISO/TC38/SC1 J.S.D.C., 69, 24 (Jan. 1953) ) were considered, and a number of new tests-for metals in the dyebath and fastness to acid chlorination, chlorite bleaching, and milling-were thoroughly discussed. The grey scale worked out jointly with the Society and also the blue scale for light fastness testing, which is almost identical with B.S. 1006: 1953, were put into force. Finally, a resolution was passed requiring the immediate introduction of the E.C.E. "code" of test methods as standard in all member countries, to replace all existing standards. The meeting felt that a considerable time must elapse before the LS.O. methods could achieve world validity, but, when that occurred, they would be adopted as E.C.E. test methods.

#### OBITUARY NOTICE

Charles W. Bancroft

Charles W. Bancroft died suddenly, at the age of sixty-three, on 10th February 1953. He joined the firm of J. Mandleberg & Co. Ltd., Salford, in 1907 as a junior in the dyeing and finishing department. Subsequently he became general manager of the company. He was appointed a director in 1938 and managing director in 1943. During his time, and under his active influence, the dyeing and finishing of proofed cloths was greatly extended by his firm, and as a result higher standards were attained throughout the industry. Although his training was purely commercial, his outlook was

one of keen interest in the technical side of dyeing and finishing, coupled with a high respect for technical and scientific attainment in others, and every wish to see it extended. He took very little active part in matters connected with the Society, but both he and his firm have always given every encouragement to their staff to become members. Such assistance at the highest business level can be a very useful source of recruitment to the Society. He was a keen sportsman and a golfer with a useful handicap. His cheerful company will be missed by a great many friends in the dyeing and allied trades.

J. W. Reidy

### New Books and Publications

#### Internationale Bibliographie der Farbenlehre 1940-1949

Folge I

By Manfred Richter. Pp. xv + 244. Göttingen: "Musterschmidt" Wissenschaftlicher Verlag, 1952. Price, DM 27.00.

The restrictions imposed on the free interchange of scientific periodicals during the war and subsequently have led to the unwarranted neglect of many interesting papers on colour science which appeared during this period. Dr. Richter's bibliography is intended to cover the whole international field of colour publications during the ten years 1940-1949, and so ensure that no important publications are overlooked. The book is purely and simply a bibliography, with the papers arranged in alphabetical order of authors' names with crossreferences. In the majority of cases only the title of the paper is listed, but in certain instances a general indication of the contents of the paper is also included. The book is completed by a helpful subject index and by a short glossary of the more important German colour terms. The typography is very clear and the general layout of the matter excellent, while the binding of the book is probably

A book of this type is, of course, entirely devoid of literary or scientific merit, and can be assessed only in terms of its completeness, especially in view of the avowed intent of the author. Confining attention solely to British publications, the present reviewer found three fairly serious omissions-W. S. Stiles, Proc. Phys. Soc., 56, 329 (1944), on "Current Problems of Visual Research"; H. G. W. Harding, J. Sci. Instrum., 25, 333 (1948), on "A Blue Glass for Approximate Colour Temperature and Intensity Measurements", and B. H. Crawford, Proc. Roy. Soc., B 134, 283 (1947), on "Visual Adaptation in relation to Brief Conditioning Stimuli". In addition papers by Hartridge (1949), Preston (1943), Straker and Vernon (1943), Weale (1949), and Clarkson, Davies, and Vickerstaff (1948) are not recorded. A number of typographical errors in the years, volumes, or pages of the references were also noted. Thus it is not possible to give this bibliography a full 100%

rating. Nevertheless, the reviewer would not like to suggest that it is unsatisfactory, and it probably rates about 98%. After all, the book does contain 1668 references (more correctly 1667, since No. 555 and 558 are the same), and as such it represents an invaluable reference book for the research worker in colour science which can be most strongly recommended.

T. Vickerstaff

#### The British Wool Manual

Pp. 543. Manchester and London: Harlequin Press, 1952. Price, 50s, 0d.

This comprehensive work, which has been written by a team of experts, covers a wide field of wool textile production, from the origin and development of the domestic sheep and goats, through the manifold diversities of processing the wide range of these animal fibres into the dyed and finished knitted or woven fabrics. In addition, some information is given on the dyeing of wool unions. The final section is concerned with the treatments for imparting to animal fibres and fabrics immunity from attack by insect pests.

In this book of some 540 pages, it may seem impossible to explain in detail each particular section of the industry, but the matter contained therein is expressed in such a lucid and concise manner that it offers a wealth of up-to-date information in its crowded pages. This information is augmented by a multitudinous list of references, which enables the reader who desires to seek further knowledge to satisfy his or her particular needs.

In this short review, it is impossible to comment on all the processes that wool undergoes from the animal's back to the finished state, so these can be discussed only in a general way.

Briefly, the first section is devoted to the various classes of wool and hair fibres, their visual characteristics and uses, and their preparation for industrial purposes. This is followed by a résumé of the modern conception of the composition and chemical, physical, and surface properties of the wool fibre. As described, these form vital links in considering their behaviour during mechanical processing and

wet treatments with a wide range of reagents and conditions normally met in practice.

The conversion of fibres into yarns is dealt with most admirably and covers the standard sequences of processing, together with useful machine data in the making of intermediate products prior to the spinning of various types of woollen and worsted yarns. Explanations are given concerning the innovation of "superdraft" and centrifugal spinning for worsted yarns, developed recently in this country; also the new American system of drawing tops into rovings in preparation for spinning.

A section on woollen and worsted weaving deals with cloth structure from the comparatively simple weaves to their more complex derivatives and the incorporation of colour and design into speciality cloths. The principles of weaving and looming are explained in detail, enabling the reader to understand the mechanisms of the different types of looms and their ancillary devices, so essential to the effective control of the warp and weft in the manufacture of superior products.

The knitting industry is represented in the making of fabrics and garments to meet present-day needs, these being constructed and fashioned in different stitches on the hosiery, weft-knitting, and warp-knitting machines, which are also described.

About one-third of the book is devoted to the dyeing and finishing of wool and wool union fabrics, these sections being very well covered. The section

on finishing is most valuable, because this topic is not dealt with in any other modern textbook.

Unfortunately, a few minor errors are included in the text; e.g. on p. 259, in the misapplication of the term "beck" used in connection with the Bradford warping mill, this should read heck.

The reviewers disagree with the statement on pp. 428–429 that in wool–cellulosic union dyeing: "Neutral dyeing acid dyes which dye cellulose fibres almost as deeply as wool should be avoided, if possible; this applies to Coomassic Red PGS". In any case, there is a contradiction, as this dye is recommended as a suitable component in the same paragraph. Two omissions occur to the reviewers—the production of felts, both woven and press, consumes substantial quantities of wool, and so do the carpet and pile-fabric industries. A short account of these processes would add to the value of the book.

These minor errors and omissions will receive due attention, no doubt, in subsequent editions. They do not detract in any way from the value of the mass of data given.

This comprehensive manual is well presented, and handsomely illustrated with innumerable diagrams and plates, and will prove to be a valuable contribution to textile literature. In addition, it presents an excellent reference book for all who care to delve into the complexities of the raw materials and their passage through the various processes into the finished state.

H. Beevers R. L. Kitchen

## **Abstracts from British and Foreign Journals and Patents**

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

#### I-PLANT; MACHINERY; BUILDINGS

Fat Extraction by Vibration. H. J. Sanders. Ind. Eng. Chem., 45, 11A, 13A (March 1953).

Four large-scale plants are now in operation based on the Chayen process, in which a slurry of bone meal (1) and water (7) is subjected to a rapid succession of shock waves, generated in a modified hammer mill. The impulses remove, almost instantaneously, 98°, of the fat, which floats to the surface and is skimmed off, whilst the extracted bone is removed simultaneously by a screw conveyor. The process avoids the degradation associated with hot rendering and the flammability hazards of solvent extraction, and produces better-quality products. It is adaptable to the extraction of fats from other materials. W. K. R.

Heat Treatment of Nylon. Anon. Teintex, 18, 169-173

A description of a stenter frame, "France IR2", manufactured by Saint-Genis & Cie. for infrared treatment of nylon fabrics.

B. K.

PATENTS

Wool-scouring Machines. P. Ward. BP 690,593
The perforated grids in scouring bowls, through which dirt and sand fall, are so arranged that they rise and fall within the bowl, thus preventing elogging and obviating the need for periodical cleansing.

J. W. B.

Thread-storing, Thread-advancing Device. H. von Kohorn. BP 690,699

Thread-storage, Thread-advancing Reels. Courtailds.

Transversely Stretching and Guiding Sheet Material.

American Viscose Corpn.

BP 690,835

Desizing Machine. Robbins Mills.  $USP\ 2.612.770$ The fabric passes in loops through the liquor, means being provided to keep the loops distended and so ensure

being provided to keep the loops distended and so ensure exposure of the entire surface of the fabric to the liquor, C. O. C.

Fallers for Stenter Frame Clips. Heberlein & Co. BP 691,407

A faller is described which, when the jaws become worn, can be easily adjusted without being reconditioned. Satisfactory gripping is attained even when the clips have not the close tolerance hitherto necessary. C. O. C.

Stretching and Guiding Tubular Fabric. J. Dungler, BP 690,283 A machine in which the internal guides can be readily

A machine in which the internal guides can be readily re-aligned or re-adjusted while the fabric is passing through it.

C. O. C.

Drive for Brushing Rollers in Teaseling Machines.
A. Monforts.

Simple means for ensuring that the brush rollers have absolutely equal circumferential velocity.

C. O. C.

Coating Flexible Sheets. W. T. Snyder. USP 2,614,522 High-speed application of a thin and even coating of a liquid is described. C. O. C.

Revolving Multiple Colour Screen Printing Table.

Multiscreen Corpn.

USP 2.613.595

Apparatus in which one operation of the squeegee transfers the colour from several screens on to the corresponding number of fabrics. C. O. C.

Dry Cleaning Apparatus. United States Hoffman Machinery Corpn. BP 690,874 Automatic control of drying in a low-temperature dry cleaning process is described. C. O. C. Extracting Cleaning Solvent from Dry-cleaned Articles. Isaac Braithwaite & Son Engineers Ltd. BP 691,467

Articles which have been dry-cleaned are removed from the cleaning solvent and placed inside a perforated cage rotating inside a fixed casing; the cage rotates to remove most of the solvent by centrifugal action, after which hot nir, provided by a heating source located between the cage and casing, is blown through the articles to remove the remaining solvent. Steam is injected for a short time to enable the fibres to recover a natural regain, and the speed is finally reduced to provide a tumbling action to open out the material. J. W. B.

Dissipating Electrostatic Charges in Textile
Machinery. T.M.M. (Research). BP 689,849
One or more parts of the machine which touch the

material being processed are coated with a composition including a radioactive agent which emits either a or  $\beta$ particles, e.g. uranium oxide or polonium 210. C. O. C.

Coating Foil. Standard Telephones & Cables

BP 690,692 A coating of varying thickness is obtained by applying an electrostatic field between a particular portion of the coated material and an adjacent electrode, of strength such that the liquid coating material is drawn toward that portion of the material. By this means it is possible to vary the thickness of the coating from place to place on the material, to free part of the surface completely from coating, or to retain the coating on an edge from which it tends to retract during drying. C. O. C.

Applying Transfers to Ceramic Blanks. British United BP 691,354 Shoe Machinery Co.

Producing Wrinkle Texture on Non-wrinkling Films (V p. 264). Dry Cleaning (VII p. 266).

#### II— WATER AND EFFLUENTS

Water-softening and -conditioning Problems— Solution by Chemical Equilibrium Methods. D. H. Caldwell and W. B. Lawrence. Ind. Eng. Chem., 45, 535-548 (March 1953).

The construction and use of water-conditioning diagrams, designed to simplify equilibrium calculations as applied to water softening, are described. Use of the graphs allows the determination of equilibrium concentrations of Ca and Mg, total alkalinity, and pH in relation to dosage with the common conditioning chemicals. Examples are given illustrating their application to typical problems, and the correlation of graphical and experimental results is discussed.

#### III- CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Fine Structure of Starch Granules. K. H. Meyer and R. Menzi. Helv. Chim. Acta, 36, 702-708 (April 1953). At temp. up to 150°c., the swelling of potato and maize starch granules is diminished, although there is an increase in dextrinisation. This is attributed to the growth of small crystallites at the expense of the free molecules. The granules become entirely sol. at 170°c. The crystallites include amylopectin as well as amylose, and it is confirmed that the outer membrane of the granules consists mainly of amylopectin. H. E. N.

PATENTS

Guanidine Alkylarylsulphonates Detergents. Lever

Brothers & Unilever. Compounds of formula  $R^1 \cdot R^2 \cdot SO_3(NH_3 \cdot C(:NH) \cdot NH_2)$   $R^3 = Alk$  of 6–18 C;  $R^2 = subst.$  or unsubst. benzene nucleus) are non-hygroscopic detergents, and do not tend C. O. C.

Detergent. American Cyanamid Co. BP 690,821 Mixtures of 98-50°, by wt. of a salt of an alkylbenzene-RP 690 821 sulphonic acid containing a single long Alk of 10-16 C. with or without one or two other Alk containing I or 2 C. and 2-50% of a water-soluble guanidine salt are good detergents for wool. Thus the presence of 10-40% of guanidine salts increases the detergency of the alkylbenzenesulphonates on wool 5-6 times, although the guanidine salts themselves have little or no detergency C. O. C.

Carding Composition for Cellulose Fibres. Monsanto. BP 689,640

A silica aquasol of pH 8-11, containing an organic hygroscopic agent (15-75%) on the wt. of the silica) and either a water-soluble emulsifying agent which is a fibre lubricant or a mixture of a water-soluble emulsifying agent and an oil lubricant (15-75), is used in such amounts as to deposit 0·1-3·0% silica on the fibres. This improves the interfibre friction of the fibres and the tensile strength of the yarns spun from them. The agent does not dust off during processing. C.O. C

BP 691,346 Textile Lubricants. Textile Oils. Lubricating compositions for textiles and textile machinery, comprising not <10% mineral oil blended with saponifiable fatty-ester oils, are mixed with 2-50% of an extremely large class of compounds of the types triesters of erythritol and pentaerythritol, some of the OH groups of which are esterified with long-chain fatty acids; ester-ethers of erythritol and pentaerythritol; and partial esters, ethers, and ester-ethers derived from tetrahydric, pentahydric, and hexahydric aliphatic alcohols of higher mol. wt. than erythritol. Such oils are readily emulsified by detergents with or without sodium carbonate.

J. W. B. Textile Lubricants. Celanese Corpn. of America

BP 691,559 A composition which serves both as a lubricant and as a conditioning agent, suitable for rendering textile fibres amenable to processing, particularly for continuous-filament rayon prior to being cut into staple, comprises a blend with mineral lubricating oil (50-70 parts by wt.), a partial ester of phosphoric acid with a long-chain aliphatic alcohol (5-15), a tertiary amine free from OH groups (1-5-4), an alkylphenol (1-4), and an alkanolamine ester and/or amide product obtainable by the action of a hydroxyalkylamine on a long-chain aliphatic acid (10-30). J. W. B.

Yarn Lubricants. Celanese Corpn. of America.

BP 690,895 Lubricants and conditioning agents for high-tenacity regenerated cellulose yarns comprise a mahogany sulphonate, an ester, amide, or ester-amide of a hydroxy alkylamine with a fatty acid having a chain of > 7 C, and They are a di- or poly-hydric aliphatic alcohol of > 5 C. C. O. C. readily removed during scouring.

Polysaccharide Derivatives of Substituted Di-carboxylic Acids. National Starch Products.

BP 691,364

Very stable and permanent emulsions are obtained by using as the dispersing agent a polysaccharide derivative containing both a hydrophilic and a hydrophobic group. Such derivatives are obtained by treating a polysaccharide with a compound of formula-

di- or tri-methylene; R<sup>2</sup> = alkyl, alkenyl, alkoxy, aralkoxy, aralkyl, or aralkenyl of 5-18 C or acyloxy of 6-19 C) or with the corresponding acid monochloride, the remaining carboxyl radical present after the reaction being the hydrophilic group. C. O. C. being the hydrophilic group.

Iminazolidinethiones. Rohm & Haas. Heating carbon disulphide at 30-250°c, with a compound of formula R1-NH-CHR2-CHR3-NH-CHR4-CHR5-NH2 (R1  $R^2$ ,  $R^3$ ,  $R^4$ , and  $R^5=H$  or  $CH_3$ , at least one member of the pair  $R^3$  and  $R^3$  and of the pair  $R^4$  and  $R^5$  being H) yields β-aminoethyl-2-iminazolidinethiones of formula-

CHR4-CHR5-NH2 R3-CH CS R2-CH-N·R1

Bis(thionoiminazolidylethyl)-thioureas or obtained by heating 2 mol. of the above products with 1 mol. of thiourea or urea at 200-250°c. All these compounds react with formaldehyde to yield hydroxymethyl compounds and condensates which are useful for treating cellulosic textiles.

C. O. C.

#### Methylenebis-2-iminazolidones. Rolum & $H_{\rm BBS}$ , UNP 2.613.210

Methylenebis-2-iminazolidones are formed when 2 mol. of a 2-iminazolidone—

are treated with 1 mol. of H-CHO in two stages, the first stage being neutral or alkaline and the second acidic. They react with formaldehyde to give NN'-bishydroxymethyl derivatives which are especially useful for treating cellulose, e.g. to render it less hydrophilic and more crush-resistant, to alter its affinity for dyes, and to confer greater resistance to chlorine and abrasion than is imparted by hitherto known products. C. O. C.

#### Solvents for Linear Polyamides. Polymer Corpn.

Free-flowing solutions of polyamides, particularly alcohol-insoluble amides, are obtained by heating the polyamides under pressure with a mixture of water (75–5% by vol.) and one or more saturated aliphatic monohydric alcohols of 1–5 C (25–95).

Solvents for Nylon. Eastman Kodak Co. USP 2,614,999 Clear, smooth, stable solutions of nylon can be prepared at room temperature by mixing the nylon with a mixture of 85% phosphoric acid (40-50%) and a liquid aliphatic alcohol, ketone, ether, or ether–alcohol of <6 C (60-50%). C. O. C.

#### Water-repellent Composition. General Electric Co.

 $USP\ 2.612.482$  Improved water-repellency of textiles is obtained by using a mixture of (a) a urea– or melamine–formaldehyde condensate, (b) a liquid organopolysiloxane, and (c) a liquid hydrolysis product of a methyldihalogenosilane of the type described in  $USP\ 2.386.259$ . C. O. C.

Unsaturated Aliphatic Phosphoramidates—Flame-proofing Agents. Glenn L. Martin Co. BP 691,358 Compounds of formula  $(R^1 \cdot O)_2 PO \cdot NR^2 R^3$   $(R^1 - Alk$  of  $3 \cdot 5$  C;  $R^2 = H$ , Alk of  $1 \cdot 3$  C, hydroxymethyl, or an unsat. aliphatic radical of  $3 \cdot 5$  C;  $R^3 = H$ , an unsat. aliphatic radical of  $3 \cdot 5$  C;  $R^3 = H$ , an unsat.  $m = 1 \cdot 5$  are useful for flameproofing textiles. C. O. C.

Ultraviolet Radiation Absorbers. General Aniline. USP 2,614,940

Compounds of formula-

(one R = H and the other = alkali-metal sulphonate radical) yield clear films when mixed with an aq. soln. at pH 5-9-5 of a plastic, and are very useful for coating materials which are degraded by exposure to sunlight or ultraviolet radiation, e.g. regenerated cellulose film.

Suspensions of Polymeric Chlorotrifluoroethylene (V p. 264).

Chemistry of Diammonium Phosphate (VIII p. 267).

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Incidents during Forty-three Years of Chemical Research. [Preparation of Phenols and Diazotisation.] H. H. Hodgson. Chem. and Ind., 236-237 (14 March 1953).

A lecture in which Dr. Hodgson dealt mainly with some fundamental discoveries with which he had been associated — (1) a general method for preparing steam-volatile phenols from aryldiazonium sulphates, and (2) a universal method of diazotisation. Arising from the latter, studies

of the mechanism of the Sandmeyer reaction were made; recent work by Cowdrey and Davies in this connection is criticised.

J. W. D.

Absorption Spectra of Polyazo Compounds. H. Dahn and H. von Castelmur. Helv. Chim. Acta, 36, 638–645 (April 1953).

Absorption spectra at 200–700 mµ, of several polyazobenzenes, substituted polyazobenzenes, azodiphenyl derivatives, and azoxy cpd. in alcohol or tetrahydrofuran are reported. Spectra of some of these cpd. in H<sub>2</sub>SO<sub>4</sub> are also reported. H. E. N.

Relation between the Absorption Spectra and the Chemical Constitution of Dyes, XXVI—Effect of Solvent and of Temperature on the cia-trans Isomerisation of Azo Dyes, W. R. Brode, J. H. Gould, and G. M. Wyman, J. Amer. Chem. Soc., 75, 1856–1859 (20 April 1953).

Study of the photochemical  $trans \rightarrow cis$  isomerisation of ten typical azo dyes in five solvents of different polarities showed that, on the whole, hydrocarbon solvents favoured isomerisation, while ethanol seemed to suppress it most extensively. Heating benzene solutions of >20 azo dyes to 65°c, shifted the isomerisation equilibrium toward the stable (trans) form whenever possible. The main absorption band at 65°c, appeared at a slightly shorter wavelength than at room temperature, but apparently only azo compounds containing one or more auxochromic substituents show this hypsochromic shift on heating.

Azoic Dyes. I — Synthesis of Fast Violet B Base. K. Konishi and A. Nishiura. J. Soc. Org. Synthet. Chem. (Japan), 10, 542-545 (1952); Chem. Abs., 47, 4085 (25 April 1953).

2-Amino-4-chlorotoluene was condensed in Na<sub>2</sub>CO<sub>3</sub> with p-toluenesulphonyl chloride at 95 c. to 2-p-tolylsulphonamido-4-chlorotoluene, converted with 15%, HNO<sub>3</sub> at 80 c. to 2-p-tolylsulphonamido-4-chloro-5-nitrotoluene, detosylated with 80%, H<sub>2</sub>SO<sub>3</sub> at 110–145 c. to 2-amino-4-chloro-5-nitrotoluene (1), methoxylated with CH<sub>2</sub>OH-KOH at 130 c. to 2-amino-4-methoxy-5-nitrotoluene, benzoylated in toluene by boiling with benzoyl chloride on an oil bath to 2-benzamido-4-methoxy-5-nitrotoluene, and finally reduced by boiling on a water bath with Fe dust (in alcohol) and 5%, HCl to 2-benzamido-4-methoxy-5-aminotoluene (Fast Violet B base)—yield > 80%, with Raney Ni catalyst > 90%. From I by similar benzoylation and reduction was obtained 2-benzamido-4-chloro-5-aminotoluene, which gave a fast brown with azoic coupling components. C.O.C.

Disazo Dyes derived from 2:3-Dimethylbenzothiazolium Salts. H. Wahl and M. T. Lebris. Compt. rend. Acad. Sci. Paris, 235, 1405-1406 (1952); Chem. Abs., 47, 3567 (10 April 1953).

A crystalline compound corresponding to a tautomer of 3-methyl. 2- (phenylazomethyl) benzothiazolium—methosulphate and 3-methyl.2-(phenylhydrazonomethyl) benzothiazolium methosulphate (I methosulphate) was obtained by heating an alcoholic solution of a 2:3-dimethylbenzothiazolium salt (II) with diazoaminobenzene. I chloride was produced directly from I or by transforming II to the azomethine (III) with p-nitrosodimethylaniline and treating III in HCl with C<sub>8</sub>H<sub>2</sub>·NH·NH<sub>2</sub>. I coupled in pyridine with C<sub>8</sub>H<sub>2</sub>·N,Cl to form 3-methyl-2-(bisphenylazomethylene)benzothiazoline, identical with that obtained by direct diazo coupling of II. III and p-NO<sub>2</sub>·C<sub>8</sub>H<sub>4</sub>·NH·NH<sub>2</sub> gave the p-nitro analogue of I nitrate, which coupled with p-NO<sub>2</sub>·C<sub>8</sub>H<sub>4</sub>·N<sub>2</sub>·Cl to produce 3-methyl - 2- (bis · p · nitrophenylazomethylene)benzo-thiazoline. C. O. C.

Thiazoles. X — Disazo Dyes from 2:2'-Diamino-5:5'-dithiazoles and Experiments on the Benzidine-like Rearrangement of 4:4'-Unsubstituted 2:2'-Hydrazothiazoles. H. Beyer and A. Kreutzberger, Chem. Ber., 85, 333-337 (1952): Chem. Abs., 47, 1697 (25 Feb. 1953).

2:2'-Diamino-5:5'-dithiazoles

(R = H, CH<sub>3</sub>, or C<sub>4</sub>H<sub>5</sub>), obtained by rearrangement of the corresponding 2:2'-hydrazothiazoles, may be tetrazotised and coupled with azo coupling components to give disazo dyes. Thus 2:2'-diamino-5:5'-dithiazole (R = II), tetrazotised and coupled with 2 mol. of naphthionic acid, gives a violet.

Attempts at a benzidine-like rearrangement of 5:5'-dimethyl(or diisopropyl)-2:2'-hydrazothiazole were unsuccessful.

Formazyl Complexes. II—Coupling Products of Diazotised o-Aminophenols and o-Carboxy-benzoylacetic Acid. R. Wizinger and H. Herzog. Helv. Chim. Acta, 36, 531-536 (March 1953).

In GP 416,379 a dye is prepared by coupling diazotised 4-chloro-2-aminophenol with o-carboxybenzoylacetic acid. It is shown that the product does not possess the dihydroxyazo structure but is a formazyl deriv. (cf. J.S.D.C., 66, 65 (1950)). It forms violet to blue complexes with Cu. Ni, or Co, green complexes with Cr. The analogous cpd. from cyanoacetic acid is also prepared. H. E. N.

Action of Acyl Cyanides on 2- and 1:2-Substituted Indoles—I. A. K. Kiang and F. G. Mann. J.C.S., 594–602 (Feb. 1953).

Acetyl and benzoyl cyanides react with 2- and 1:2substituted indoles in the presence of HCI usually to give bis-3-indolyl derivatives of type I, whereas in the presence of pyridine the 3-acylindole is formed. Compounds of type I lose HCN when boiled with nq--alcoholic KOH, yielding ethylenic derivatives which give deeply coloured salts of suggested types II (cationic) and III (anionic).

Chemical Constitution and Photographic and Allergenic Properties of Colour Developers. A. Weissberger, Colloque Sensibilité phot. Paris (1951), Science et Inds. phot., 23 A, 238-242 (1952).

Quinones. II—Addition of Mercapto Acids to Benzoquinones and 1:4-Naphthaquinone. A. Blackhall and R. H. Thomson. J.C.S., 1138-1143 (April 1953).

Theory of Metachromasis. M. Mizushima. Busiciron Kenkyu (Researches on Chem. Phys.), (17), 38–40 (1949); Chem. Abs., 47, 3694 (25 April 1953).

Metachromasis is the change in colour of a dye soln, when a polymeric ion is added. Perturbation calculation is made of the shift of absorption frequency in the case of Methylene Blue and polyvinyl sulphate on the assumption that a dye mol. is adsorbed by an ionic group of the polymer mol. and that no appreciable changes in electronic state occur, so that ion-dipole interaction is treated as a perturbation potential.

C. O. C.

Effect of Methylene Blue on the Oxygen Consumption of the Mitochondria of Liver. F. Leuthardt and B. Exer. Helv. Chim. Acta, 36, 519-526 (March 1953).

The dye appears to have two superimposed effects—
(i) acceleration of respiration and (ii) irreversible damage
to the mitochondria, resulting in inactivation of the
respiratory system.

H. E. N.

Thermochromism and Keto-Enol Tautomerism of Solutions of 1:3-Diketo-2-phenyl-5-bromoindane. A. Schönberg, A. Mustafa, and W. Asker. Nature, 171, 222 (31 Jan. 1953).

Simple Synthesis of Coloured Flavylium Salts. R. Wizinger and A. Luthiger. Helv. Chim. Acta, 36, 526–530 (March 1953).

Several new coloured flavylium salts are prepared by

condensing flavone in presence of POCl<sub>2</sub> with dimethylaniline, diarylethylenes, or p-dimethylaminocinnamic acid, and, more readily, by warming flavylium perchlorate with the same cpd., when condensation is accompanied by dehydrogenation.

H. E. N.

Usnic Acid. IX—A Revised Structure for Usnolic Acid and the Resolution of (±)-Usnic Acid. F. M. Dean, P. Halewood, S. Mongkolsuk, A. Robertson, and W. B. Whalley. J.C.S., 1250–1261 (April 1953).

Carotenoid Syntheses. XI—Further Polyenes related to 1:8-Diphenyl-3:7:12:16-tetramethyloctadecanonaene. C. F. Garbers, C. H. Eugster, and P. Karrer. Helv. Chim. Acta, 36, 562-569 (April 1953).

Rhodopsin and Indicator Yellow. F. D. Collins, Nature, 171, 469-471 (14 March 1953).

Irradiation converts rhodopsin (visual purple) into retinene, which is further changed into indicator yellow. The marked alteration in spectral absorption which results when a soln, of indicator yellow is varied in pH is paralleled by the behaviour of vitamin A aldehyde in presence of aliphatic amines, and this suggests that indicator yellow is a compound of retinene with a free amino group. Evidence is presented, based upon spectral absorption with changing pH, and measurement of re-action rate, that alkaline indicator yellow is a Schiff's base; the acid form is believed to be more complex, and is probably a double molecule. A carbon-nitrogen bond is present in rhodopsin, and does not result in indicator vellow from the combination of retinene with free amino groups such as those in proteins which may happen to be present when it is formed; it is not, in fact, a fortuitous artefact. Retinene methylimine appears to be a close analogue of indicator yellow. Any further knowledge of the chemistry of rhodopsin now depends on the elucidation of the structure of indicator yellow. J. W. D.

Short-cycle Syntheses of Ultramarine Blue. C. A. Kumins and A. E. Gessler. Ind. Eng. Chem., 45, 567-572 (March 1953).

By calcination of potentially zeolite-forming substances in the presence of organic sodium salts and sulphur, ultramarine blues of superior tinetorial strength were obtained in time cycles of about 7 hr. as compared with days in the normal process. Three new syntheses were developed, based on sodium aluminate-silica mixtures, calcined causticised kaolin, and precipitated zeolite. The chemistry of both the reduction and conversion step was studied, and data were obtained which explained the rapidity of the reaction; it was also shown that the "oxidation" step involved the removal of sodium oxide from the crystal lattice. X-Ray diagrams indicated that all ultramarines, irrespective of the method of formation, were identical, though the intermediate products were not. W. K. R.

Absorption Spectra of Co-ordination Complexes. V—The Hyperchromic Series of Ligands. 8. Yamada and R. Tsuchida. Bull. Chem. Soc. Japan, 26, 15-19 (Jan. 1953).

The extinction coefficients of the first band of the absorption spectra of cobaltic complexes decreases as the ligand is changed in the order—NCS , CrO<sub>4</sub><sup>2</sup> , SO<sub>5</sub> , NO<sub>2</sub> , O<sub>7</sub><sup>2</sup> , en, OH , NO<sub>5</sub> , SO<sub>4</sub><sup>2</sup> , Br , NH<sub>3</sub>, Cl , H<sub>2</sub>O. For cobaltammine complexes containing ligands co-ordinated through oxygen (e.g. nitroammino-cobaltic complexes) ν<sub>max</sub> is linearly proportional to log ε<sub>max</sub> . A. J.

PATENTS

Red Metachrome Monoazo Dyes. Gy. BP 690,600 Monoazo compounds—

$$\begin{array}{c|c} O_2N \text{ OH} & \begin{matrix} O \\ C \end{matrix} & X \\ \hline N=N-CH & N- \\ R & \begin{matrix} HOOC \cdot C \end{matrix} & X \\ \end{array}$$

(R = alkyl or cycloalkyl of 5–8 C; X = H, or one X = H and the other = Cl) are red metachrome dyes and possess better affinity from a neutral bath than the similar dyes of BP 561,228 (J.s.b.c., 60, 249 (1944)), which contain a sulpho or two carboxyl groups. Thus 2-amino-4-cyclohexyl-6-nitrophenol is diazotised and coupled with an

C. O. C.

alkaline solution of 1-m-chlorophenyl aparazolone-3carboxylic acid. The resulting monoazo dye is preferably mixed with 10% of its weight of soda ash and a dispersing agent, and is a bluish-red metachrome or after brome dye.

#### Fluoroalkyl Monoazo Disperse Dyes from 2:4-Bismethylsulphonylaniline. Eastman Kodak Co. USP 2,615,013

Monoazo compounds obtained by diazotising 2:4-bismethylsulphonylaniline or its 6-chloro or 6-bromo derivative and coupling with-

$$\bigvee_{\mathbf{X}}^{\mathbf{Y}} \mathbf{N} \mathbf{R}^{1} \mathbf{R}^{2}$$

 $\begin{array}{lll} (R^1=CH_2\cdot CHF_2,\; CH_2\cdot CF_3,\; CH_2\cdot CH_3,\; CH_2\cdot CHF_2,\; CHF_2,\; CHF_2\cdot CHF_2,\; CH_2\cdot CH_2\cdot CH_2\cdot CH_3,\; CH_2\cdot CH_2\cdot CH_3\cdot CH_2\cdot CH_3\cdot CH_2\cdot CH_3\cdot CH_$ sulphonylaniline in nitrosylsulphuric acid, and coupling with an acid solution of  $N \cdot \beta \beta$ -diffuoroethyl  $N \cdot \beta$ -hydroxy-ethylaniline, adding soda ash to neutralise and complete the coupling, a monoazo dye giving orange on cellulose acetate is obtained.

#### Monoazo Disperse Dyes for Cellulose Esters. Eastman USP 2,615,014 Kodak Co

Monoazo disperse dyes-

(R<sup>1</sup> =  $\gamma$ -hydroxypropyl,  $\beta \gamma$ -dihydroxypropyl, or preferably  $\beta$ -hydroxyethyl; R<sup>2</sup> =  $\beta \beta$ (or  $\gamma \gamma$ )-diffuoro-n-propyl, ββ(or γγ)-difluoro-n-propyl, ably  $\beta$ -hydroxyethyl;  $R^* = \beta\beta(\sigma^* \gamma \gamma)$ -diffuoro-n-propyl,  $\gamma \gamma$ -diffuoro-n-butyl,  $\delta \delta$ -diffuoro-n-amyl, or preferably  $\beta \beta$ -diffuoroethyl; X = H, Br, or Cl; Y = H, F, Cl, Br,  $SO_2 CH_3$ ,  $CF_3$ , or, where X = H, Y may  $= SO_2 NH \cdot CH_3$  or  $SO_2 NH \cdot C_2H_5$ ; Z = H, Br, Cl,  $CH_3$ , or  $C_2H_3$ ) give oranges to rubines of good light fastness on cellulose esters. Thus, 2-amino-5-nitrophenyl methyl sulphone is diazotised in nitrosylsulphuric acid and coupled with  $N\!-\!\beta\beta$  -diffuoroethyl- $N\!-\!\beta$  -hydroxyethylaniline to yield—

$$NO_{2} \underbrace{-N:N-}_{SO_{2} \cdot CH_{3}} \underbrace{-N:CH_{1} \cdot CH_{2} \cdot CH_{2}$$

which dyes acetate rayon scarlet.

### Monoazo Pigment Lakes for Synthetic Resins. ICL

The metal (especially Ca and Sr) salts of the monoazo compounds 4-chloroanthranilic acid → 3-methyl-1-phenyl-(or tolyl)-5-pyrazolone are suitable for incorporation into synthetic resins for giving transparent yellows

BP 691,482 The metallic lakes, which may be rosinated, of the monoazo compounds aniline - 2 · sulphonic acid(or its 5-chloro derivative)  $\rightarrow$  2-hydroxy-3-naphthoic acid are suitable for incorporation into synthetic resins for giving transparent reds.

#### Azoic Dyes—Preparation of Diazoamino Com-pounds. Fran. BP 691,410 pounds. Fran.

Diazo compounds derived from aromatic diamines, in which one of the amino groups is secondary and also carries a sulpho group, combine with simple secondary amines to give soluble diazoamino compounds. Hot dilute acid treatment regenerates the diazo compound and eliminates the sulpho group, so that these products may be used in the production of azoic dyeings and prints. Thus N-p-methoxyphenyl-4-diazobenzenesulphamic acid, in the form of its internal salt, prepared according to the method of BP 611,320 (J.S.D.C., 65, 186 (1949)), is condensed with diethylamine in presence of soda ash. The soluble diazoamino compound

$$\begin{array}{c|c} CH_{2} \cdot O & -N \cdot N \cdot N (C_{2}H_{5})_{2} \\ \hline \\ SO_{3}Na & \end{array}$$

is isolated by salting out. It is mixed with 2-hydroxy-3naphthanilide and sodium hydroxide solution, thickened, and printed on cotton. After drying, the blue azoic dye is developed by steam containing acetic or formic acid.

# Triphendioxazine Sulphur Dyes. DuP. USP 2,600,690

Violet sulphur dyes of general formula-

 $(Z=Cl\ or\ Br;\ R=CH_3\ or\ C_2H_5;\ X=sulphide-vattable group,\ e.g.\ SCN,\ alkyl\ xanthate,\ or\ S-8\ linking\ another$ dioxazine residue) are made by tetrazotising the corresponding diamine (X = NH<sub>2</sub>) and treating with an alkalimetal thiocyanate, xanthate, or disulphide. The starting material is prepared e.g. by condensing dianisidine (2 mol.) with chloranil (1 mol.).

#### Red Iron Oxide Pigments. Kali-Chemie. BP 691,457 Hot air is blown into a solution of a ferrous salt in presence of metallic iron and, as inoculating agent, ferric oxide, and the resulting red oxide filtered, washed, and dried at 80-120 c. The products are very soft, and, since the initial oxide is light-coloured and darkens by growth as the process continues, reds of any desired depth can be

Flow of Concentrated Suspensions (V below).

Physicochemical Properties of the Surface of Aqueous Solutions. IV Classification of the Mechanical Properties of Several Aqueous Surfaces (VII p. 265).

Physicochemical Properties of the Surface of Aqueous
Solutions. V— Mechanical Properties of the Surface of Dye Solutions (VII p. 266).

#### V-PAINTS; ENAMELS; INKS

#### Flow of Concentrated Suspensions. P. 8. Williams.

J. Appl. Chem., 3, 120–128 (March 1953).

The paper is in two parts, of which the first deals with the general properties of the flow of cone, suspensions, and the shear stress rate of shear characteristics of Newtonian, non-Newtonian, plastic, and thixotropic types of flow. The types of suspensions encountered in the paint industry and their flow properties are described. No generalisation is yet possible on the effect of particle size or shape or the nature of the chemical interface. In the second part, an account is given of recent research on the flow of glass spheres in simple liquids. The particle-size distribution greatly affects the viscosities of suspensions with > ca.  $30^{\circ}_{\circ_i}$  of spheres by vol. The nearer particle sizes are to uniformity, the lower is the conen. of spheres required for a given viscosity. For particle sizes  $< 1~\mu$ . in diameter,

#### J. W. D. Co-operative Research—Exposure Testing on a Co-operative Basis. A. Pass. J. Oil & Col. Chem. Assocn., 36, 151-170 (April 1953).

the smaller the size, the greater the thixotropy.

An account of the problems encountered in conducting a series of paint exposure tests on a co-operative basis, and of the steps taken to overcome some of the difficulties. The exposure of duplicate panels is insufficient to avoid difficulties which may arise from wood faults, and uniformly used practical standards must be supplemented by check tests with individual observers. The most practicable method for preparation is to have all panels painted at one place by a skilled painter, and distributed from there. Check tests should be carried out before and during exposure, and it is preferable to have also a con-trolling observer to visit various sites, checking the observations made by those on the spot. Each test panel should be confined to one paint system, otherwise a durable paint may have to be withdrawn before it is fully weathered, or an inferior paint may have to be allowed to reach an advanced stage of deterioration. meteorological records should be obtained at each exposure site. The methods used to handle the large mass of accumulated data are described. Eight basic recommendations are made regarding the conduct of such trials. Some of the results obtained in the tests are used to illustrate variations in behaviour of certain types of pigments when used in paint films at different places in Great Britain.

J. W. I.

#### PATENTS

 $\begin{array}{ccc} \textbf{Inks} & \textbf{for} & \textbf{Printing} & \textbf{on} & \textbf{Polyethylene} & \textbf{Film.} & Sun \\ & Chemical Corpn. & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ &$ 

An ink, which is not readily removed from polyethylene film by sudden removal of an adhesive material from it, contains as the vehicle an aromatic hydrocarbon solvent and as binder a mixture of a polyethylene resin, wax, and natural or synthetic rubber.

Powdered Pigment Compositions. Sherwin-Williams  $USP\ 2,613,158-60$ 

Pigment compositions dispersible by simple stirring in ordinary paint, enamel, or lacquer vehicles to yield full colour value are obtained by milling resins with pigments. The proportion of resin to pigment depends upon the particular pigment used, but appears to be related to the surface area of the pigment and is greater than that required to produce a monomolecular coating of the resin on the pigment particles.

C. O. C.

Pigments having Better Wettability in Paint Vehicles and Yielding Films of Higher Gloss. DuP. USP 2,615,006

Pigments, especially TiO<sub>2</sub>, which have been treated with a small amount of a silicone polymer or an organopolysiloxane have superior gloss properties when used in oleoresinous or gloss emulsion conting compositions; in addition they are more readily wetted by relatively poor wetting vehicles, e.g. synthetic resin systems. C. O. C.

Suspensions of Polymeric Chlorotrifluoroethylene. British Thomson-Houston Co. BP 691,550

Suspensions of solid polymeric chlorotrifluoroethylene in an aqueous soln. of an aliphatic ether, monohydric alcohol, ketone, ester, lactone, or acetal or a mixture of a monohydric and a dihydric sat. alcohol are stable and are used for coating and impregnating.

C. O. C.

Producing Wrinkle Texture on Non-wrinkling Films. New Wrinkle. USP 2,613,393

Spraying water on to a warm non-wrinkling coating of a thermoplastic resin and a solvent of high evaporation rate develops a wrinkle texture of uniform pattern. A pattern can be developed in the wrinkle texture by locally leating the coating.

C. O. C.

Ornamental and Protective Crackle Coating. National Cash Register Co. USP 2.612.456

The surface, e.g. of steel plate, is first coated with a vinyl chloride-vinyl acetate copolymer dispersed in an organic liquid which readily evaporates below the m.p. of the copolymer and which has a slight swelling effect on the copolymer, and is rapidly dried below the m.p. of the copolymer; the resulting cracked coating is then heated at above the m.p. of the copolymer so as to form a continuous adherent film.

C. O. C.

Temperature-indicating Paints (XIV p. 271).

#### VI-FIBRES; YARNS; FABRICS

Electrical Resistance of Textile Materials. I—
Influence of Moisture Content; II—Effect of
Temperature; III—Miscellaneous Effects; IV—
Theory. J. W. S. Hearle. J. Textile Inst., 44, ±117—
±143; ±144-±154; ±155-±176; ±177-±198 (April

I—The electrical resistance of a large number of types of fibre is measured under different conditions of humidity, and the results are treated mathematically and expressed

as a representative equation.

II—Variation of resistance with temperature is expressed as an equation for cotton, viscose and acctate rayons, and wool, and as an approx. equation for silk, all at several moisture contents, and results are given at a single moisture content for purified cotton, ramie, and nylon.

III — Effect of voltage, initial variation of resistance with time, effect of the nature of the electrodes, effect of tension on the specimen, behaviour of composite specimens, and back e.m.f.s are studied.

IV— The two main theories of conduction by ions at fairly low moisture contents, i.e. the theory of the limitation of the movement of ions by breaks in the conduction path, and the theory of limitation of dissociation of ion pairs in a medium of low dielectric constant, are examined and developed.

J. W. B.

Moisture Gradients in Centrifuged Samples, J. M. Preston and M. V. Nimkar. Text. Research J., 23, 64-65 (Jan. 1953).

In this letter results obtained by Welo, Ziifle, and McDonald (ibid., 22, 261 (1952)), who used a freezing technique for measuring moisture gradients, are discussed in relation to results previously published by Preston, Nimkar, and Gundavda (J. Textile Inst., 42, r 79 (1952)). It is concluded that the capillary water probably lies somewhere between the very low values indicated by Ashpole (Proc. Roy. Soc., A 212, 112 (1952)) and the very high values indicated by Welo et al., and nearer to those of the present authors.

A. B.

Viscometric Studies of Cellulose in Cotton in relation to Mechanical Processing, C. M. Conrad and R. A. Rusca. Text. Research J., 23, 168-174 (March 1953).

It is shown that chemical damage is not produced in ordinary cotton textile processing to any appreciable extent. Viscosity data are employed, using statistical analysis and the Krieble and Whitwell treatment of the data.

A. B.

Degradation of Cellulose during Mechanical Processing. III—Comparison of the Evaluation by Viscometric Methods. J. C. Whitwell and R. F. Schwenker. Text. Research J., 23, 175-185 (March 1953).

It is shown by viscometric methods that there is a significant decrease in intrinsic viscosity of cotton samples which have undergone standard mechanical processing. Methods and results used in this work are discussed in comparison with work carried out by Conrad and Rusca (preceding abstract).

A. B.

Theory and Morphology of Crimped Rayon Staple.
M. Horio and T. Kondo. Text. Research J., 23, 137–151
(March 1953).

The formation of fine crimped viscose rayon staple by spinning into a bath containing a high proportion of salt and a low proportion of acid is discussed. It is shown that the crimps become finer as the alkalinity increases up to 3% NaOH, becoming coarser as the conen. increases above this, until straight filaments are produced in a 7% solution. At higher alkalinity the filaments crimp again, but in the reverse direction. The characteristic cross-section of crimped fibres is discussed in detail.

A. B.

Skin Effect in Crimped Rayon. W. A. Sisson and F. F. Morehead. Text. Research J., 23, 152-157 (March 1953).

The formation of crimp in viscose rayon staple is shown to be due to an unbalanced condition of the cross-section, resulting from an asymmetrical distribution of skin around the core. Unbalanced cross-sections may be formed from conjugate filaments, broken-skin filaments produced by chemical effects in the spinning bath, and irregular serrated filaments probably produced by the bath flow.

A. B.

Iodination of Keratin. H. R. Richards and J. B.

Speakman. Nature, 171, 751 (25 April 1953). Aqueous 0.1 N. iodine solution at 22-2 c. and pH 9 iodinates only 75% of wool tyrosine even after 64 hr. Almost complete conversion to 3:5-di-iodotyrosine is obtained, however, by treating wool (0·421 g.) with 0·78 N. iodine in absolute alcohol (50 ml.) for 72 hr. at 25%. Tyrosine contents of treated and untreated wools are 0·214 and 4·77% respectively. Similar experiments with amino acids show that histidine reacts to a negligible extent, and tryptophan and proline not at all. Partial iodination of tyrosine gives more monoiodotyrosine than di-iodotyrosine, contrary to an earlier opinion that tyrosine is iodinated slowly to the mono derivative, which then reacts instantaneously to give the di-iodinated compound. J. W. B.

Use of Calcium Hydroxide in the Preparation of Peanut Protein. J. Pominski and W. O. Gordon. J. Amer. Oil Chem. Soc., 30, 88-89 (Feb. 1953).

It was shown in laboratory peptisations that in the pH range 7:2-9-5 nitrogen solubility obtained with Ca(OH)<sub>2</sub> soln, was constant and was practically equal to the value obtained with the use of NaOH at pH 7:5. Pilot-plant yields of protein and settling rates of protein eards from Ca(OH) rectified solve with the control of the period o Ca(OH)<sub>2</sub>-peptised soln. using SO<sub>2</sub> to lower the pH were equal to those obtained previously from NaOH-peptised P. G. M.

Statistically Coiled Fibre Molecules, H. Kuhn, F. Moning, and W. Kuhn. Helv. Chim. Acta. 36, 731-752 (April 1953).

Fibre molecules can be represented as consisting of straight elements of equal length, the angle which one element makes with its neighbour varying at random. A new model is now described in which the elements are not straight but bent into segments of a circle in such a way that the direction of the tangent at the beginning of one element is statistically orientated to the tangent at the end of another element. By means of models made of wire, it is shown how equations for diffusion, sedimentation, viscosity, and streaming birefringence can be corrected to give better agreement between theory and experiment.

H. E. N Textile Fibres—Variations on Some Familiar Themes. J. R. Whinfield. Chem. and Ind., 226-229 (14 March 1953).

An account of the background to, and the development of, synthetic fibres in general and Terylene in particular. . W. D.

Separation of iso- and Tere-phthalic Acids by means of the Thallous Salts. D. Bryce-Smith. Chem. and Ind., 244 (14 March 1953)

The mixture of isomeric acids is brought into mildly ammoniacal soln., and a soln. of thallous acetate is added. Whilst the soln, is kept at 0-5°c, for some hours, thallous terephthalate separates out and may be filtered off. ino-Phthalic acid may be pptd. from the filtrate by means of H<sub>2</sub>SO<sub>4</sub>. If phthalic acid is present, its thallous salt may be separated from the other two by virtue of its ready solubility in cold water. Full details are given. J. W. D.

Imparting Controlled Crimp to Wool Fibres.

Alexander Smith & Sons Carpet Co. BP 691,391

Textile fibres are passed through a zone containing a heated fluid and moisture in which they are presoftened, and are then gripped between closely spaced conveyor surfaces discharging into a substantially closed zone in which they are held, in crimped form owing to folding, against the mass of fibres already there. By pretreatment, or by steam treatment within the closed zone, the fibres should contain 20-30% by wt. of water; they are then treated with either superheated steam or a suitable setting agent, e.g. thioglycollic acid, introduced through a number of spaced points in the path of travel, to make J. W. B. the crimp permanent.

Regenerated Keratin Filaments. Courtaulds

An aqueous solution of keratin, produced e.g. by stirring wool waste (100 g.) into 800 ml. of 98% formic acid, is treated with peracetic acid, produced in situ, e.g. by adding 5 lots each of 10 ml. of 100-vol. H2O2 and 5 lots each of 8 ml. of acetic anhydride, alternately at 30-min. intervals, after which it is stirred for 18 hr, to dissolve. Precipitation of a curd by addition of water is followed by filtration, purification, and redispersion in  $NH_4OH$ . The liquid is extruded into a MgSO<sub>4</sub>-Na<sub>2</sub>SO<sub>4</sub> aqueous bath, the thread stretched while swollen, and hardened in a formaldehyde-J. W. B. metal salt bath.

Hardening Regenerated Protein Filaments. Courtaulds.

Artificial protein filaments of improved wet strength are produced by extruding the protein, e.g. milk casein, as a dil. aq. soln, of pH > 7 into an aq. coagulating bath containing > 25 g.  $H_0SO_1$  and < 400 g.  $Al_2(SO_4)_2, 18H_2O$ per litre, possibly with Na2SO4 and formaldehyde also. J. W. B.

Esterification of Extruded Protein

Protein threads having improved wet properties are produced by hardening freshly extruded filaments in a formaldehyde-metal salt bath until they are resistant to aqueous swelling, treating them at 35-50°c, in a methyl or ethyl alcohol bath in presence of an esterification catalyst, e.g. H<sub>2</sub>SO<sub>1</sub>, and possibly a dialkyl sulphate, e.g. diethyl sulphate, and then curing the esterified threads in a second hardening bath.

Enhancing the Resistance of Nylon to Light. ICL  $BP\ 689,629$ 

The salts of phenolic acids and anthranilic acid with Cr. Al, Cu, Mn, and some other metals, when added to the nylon melt, much enhance the resistance to light of the products made from the melt.

Synthetic Polypeptide Fibres. Courtaulds. BP 690,341 Fibres are produced by spinning a copolymer of DL-β-phenylalanine and DL-leucine dissolved in methylene chloride into hot air or into a coagulating liquid, e.g. petroleum ether.

Filaments, etc. from Aqueous Dispersions of Poly-tetrafluoroethylene. DuP. BP 689,801 Aqueous colloidal dispersions of polytetrafluoroethylene, the insolubility of which in solvents precludes solutionextrusion techniques, are prepared in which at least 10% of the particles have an elongated ribbonlike shape with a width  $<0.07~\mu$ ., a length : width ratio >5:1, and a cross-sectional area  $<0.001~\mathrm{sq}$ .  $\mu$ ., the dispersion containing 15-75% of polytetrafluoroethylene by wt. The dispersing agent is an ammonium or alkali-metal salt of a polyfluoroalkanoic acid  $H \cdot [CX_x \cdot CX_x]_n \cdot COOH$  ( X = F or Cl, at least half the Xs in each  $CX_x \cdot CX_x$  unit being F; n = 3-10). After coagulation in an acid bath the filament is washed, dried, and sintered above 327 c. to coalesce the particles prior to cold drawing, leading to a highly oriented, strong, and resistant fibre. J. W. B.

Solvents for Polyacrylonitrile. Industrial Rayon BP 690,118

Addition of a small amount of a polyhydric alcohol, e.g. ethylene glycol, to a solution of an acrylonitrile polymer in ethylene carbonate decreases the viscosity of the mixture and makes it more useful for spinning fibres.

Fibre-forming Solutions of Acrylonitrile and Vinylpyridine Polymers. Chemstrand Corpn.

USP 2,613,194-5 Blended polymers of > 50% of non-dyeable acrylo-nitrile polymers and < 50% of a vinylpyridine or alkylsubstituted vinylpyridine polymer dissolve in the usual solvents for acrylonitrile polymers to produce unusually homogeneous and spinnable solutions if the solutions are made in presence of 0.01-3.0% on the wt. of soln. of sulphuric acid or 0.02-5.0% of alkyl esters of aromatic C. O. C. sulphonic acids.

Acrylonitrile Copolymers having Affinity for Acid Dyes. Chemstrand Corpn.

Copolymers of acrylonitrile and a vinyl ester of a halogen-substituted monocarboxylic acid are given affinity for acid dyes by treatment of their solutions with a trialkylamine, particularly trimethyl- or triethyl-amine.

Mixed Acrylonitrile Polymers of Improved Resistance to Heat. American Viscose Corpn. BP 691,493
Mixed polymers containing 4 90%, by wt. of acrylonitrile and 2-50%, of a copolymer of acrylonitrile or methacrylonitrile (10-70%) and a substance containing only one C.C. group (90-30%) have much improved

resistance to heat.

Flow of Concentrated Suspensions (V p. 263),

#### VII DESIZING; SCOURING; CARBONISING; BLEACHING

Physicochemical Properties of the Surface of Aqueous Solutions. IV Classification of the Mechanical Properties of Several Aqueous Surfaces, H. Kimizuka. Bull. Chem. Soc. Japan, 26, 30-33 (Jan.

The surface rigidity and surface viscosity coefficients of aqueous soln. of saponin (1), Crystal Violet (II), polyvinyl

alcohol (III), horse serum albumin (IV), gum arabic (V), peptone (VI), egg albumin (VII), and sodium stearate (VIII) have been measured, and the changes of visco-elastic properties with conen. expressed in terms of mechanical models (bid., 25, 318 (1952)). With increasing conen. of III-VIII, the models change from dashpot through Maxwell model to spring and Voigt element in series, and with increasing conen. of I, II, and VIII change from dashpot, through Voigt element, to spring and Voigt element in series.

A. J.

Physicochemical Properties of the Surface of Aqueous Solutions. V—Mechanical Properties of the Surface of Dye Solutions.— H. Kimizuka. Bull. Chem. Soc. Japan, 26, 33–35 (Jan., 1953).

The rheological properties of 10 <sup>4</sup> m, soln, of Crystal Violet (I), Night Blue (II), Congo Red (III), Phloxine (IV), Scarlet Red (V), and Methyl Orange (VI) have been studied over the pH range 1–13. 1–1V form a coherent adsorbed layer and show sharp maxima in surface rigidity and viscosity with changes in pH. The effect is considered to be due to differences in interaction between molecules in the adsorbed layer.

A. J.

Properties of Freshly Formed Surfaces. XIX— Dynamic Surface Potentials and the Desorption Process: the Spreading of Decyl Alcohol Films. C. C. Addison and D. Litherland. J.C.S., 1150–1155 (April 1953).

Properties of Freshly Formed Surfaces. XX — Use of Dynamic Surface Potentials in the Study of Adsorption Rates. C. C. Addison and D. Litherland. J.C.S., 1155-1159 (April 1953).

Properties of Freshly Formed Surfaces. XXI—Influence of Chain Length and Structure on the Surface Potentials of Aqueous Solutions of Alcohols. C. C. Addison and D. Litherland. J.C.S., 1159-1167 (April 1953).

Structure and Aggregation in Dilute Solutions of Surface-active Agents. H. B. Klevens. J. Amer. Oil Chem. Soc., 30, 74-80 (Feb. 1953).

Critical micelle concentrations (C.M.C.) are shown to depend on chain length. All straight-chain saturated surface-active agents of equal ion length have approximately the same C.M.C. Thus a C<sub>12</sub> fatty-acid soap, a C<sub>12</sub> sulphonate, a C<sub>11</sub> sulphate, and a C<sub>12</sub>-ammonium chloride have C.M.C. values of 0·010–0·014 M. Values of C.M.C. are not changed to any extent by substitution near the hydrophilic head of one, two, or three groups, even as large as hydroxyethyl, in place of the amine hydrogens in the cationic detergents. However, substitution with dihydroxypropyl groups has a marked effect on association in C12 but not in the C16 series, indicating that the relative lengths of the two chains must be considered as an important factor in association. This is well illustrated in the dialkyl sulphosuccinate series, and in a tetradecyl sulphate series in which the -SO<sub>4</sub>Na group was progressively moved down the chain, particularly when the branched-chain compounds are compared with the corresponding straight-chain detergents of length equal to the maximum length from the charged head to the ultimate carbon atom. Introduction of double bonds causes a small but definite increase in C.M.C., whereas polar substitution in the chain results in a marked increase in C.M.C. Possible micelle structures are discussed in the light of these association phenomens, and it is concluded that the assignment of a definite size and shape to micelles appears at the present time to be slightly premature. However, if one includes the concept of relative orderdisorder in the micelle as one of the factors, in addition to chain length, type of detergent, and environment, which are important in micelle structure, it is possible to explain partly the apparent marked differences which have been reported for micelles of different surface-active agents.

Detergency and Foaming Properties of the System Alkylarylsulphonate - Soap - Sodium Carboxymethyl Cellulose, T. H. Vaughn, H. R. Suter, and M. G. Kramer, J. Amer. Oil Chem. Soc., 30, 1-5 (Jan. 1953).

A study has been made of the detergency and foaming characteristics of a ternary system comprising a sodium alkylarybulphonate, a medium-titre soap, and sodium carboxymethyl cellulose under average conditions prevailing in household laundering, the experiments being carried out in a modified type of washwheel. Carbon soil removal and whiteness retention properties of soap alone are equalled or exceeded in compositions containing 5 parts of soap to 1 part of alkylarylsulphonate with a minor portion of sodium carboxymethyl cellulose. Greater foam volumes result with detergent than with soap at concn. <0.2% in water of hardness 9-6-11-4 parts per 100,000 (as CaCO<sub>3</sub>). Sodium carboxymethyl cellulose, when used with soap and alkylarylsulphonate in the proportions desirable for improvement of detergency, does not affect foaming properties significantly. P. G. M.

Wettability of Solid Surfaces. VI — Effects of Metallic Cations on Stearic Acid Built-up Films. A. Inaba. Bull. Chem. Soc. Japan. 26, 43, 49 (Jap. 1953)

Bull. Chem. Soc. Japan, 26, 43–49 (Jan. 1953). The wettability of mixed built-up films of stearic acid and Ca or Ba stearate decreases with the number of layers, the advancing contact angle tending to 115°. Increase in Ba or Ca conen. causes a decrease in wettability. Ba and Ca form stoichiometric stearates which are mobile in the surface, which slowly becomes hydrophilic. Al and Cu stearates rapidly form a molecular network, the surface becoming temporarily hydrophilic; and Hg stearate slowly forms a network, the surface becoming hydrophobic.

Soiling and Soil Retention in Textile Fibres— Suspending Power of Surface-active Agents. J. Compton and W. J. Hart. Ind. Eng. Chem., 45, 597-602 (March 1953).

The suspending power (S.P.) of surface-active agents was studied using chopped cotton fibres and an aqueous dispersion of carbon black. The highest S.P. was shown anionic cellulose derivatives (e.g. carboxymethyl cellulose) and polyvinyl alcohol. The effectiveness of the cellulose derivatives must be due to some factor inherent in the polymer structure, as similar starch derivatives have little S.P. Mixing surface-active agents usually results in synergism or antagonism, but these effects cannot be predicted. The S.P. of detergents is lowered by the addition of alkaline builders, but the effect can be overcome by the use of small amounts of synergistic highpolymer suspending agents, the amount required varying with the detergent and the quantity of builder present.
With increasing temperature, the S.P. of soap, both alone and in the presence of additives, decreased, whilst little change was noted in the S.P. of two synthetic detergents under similar conditions.

Soiling and Soil Retention in Textile Fibres—Grease Carbon Black Soil Cotton Fibre Systems; Primary Deposition of Grease-free Carbon Black Soil on Various Types of Textile Fibres. J. Compton and W. J. Hart. Text. Research J., 23, 158-163, 164-168 (March 1953).

In the first paper the authors show that the sorptive binding of soil to the fibre occurs by a grease layer. The second paper shows that there is a marked variation of the percentage distribution of total surface occupied by crevices of varying diameter. Wide variations are also found in the rate of primary deposition of soil, and the suspending power of different surface reactants. A. B.

#### PATENTS

Dry Cleaning. J. T. Lascari. USP 2,614,026
At a predetermined stage in the cleaning, finely divided water is introduced into the solvent, which already contains a detergent and an emulsifying agent. This not goods but also reconditions them.

C. O. C.

Nullifying the Inhibitory Effect on Water-repellency of Traces of Surface-active Agents on Fabrics which have been Dry-cleaned. Emery Industries. BP 689,504

Impregnation with a metallic salt of the type of aluminium acetate or an alkoxide of the type of aluminium propoxide added to dry-cleaning solvents containing detergents prevents any reduction in the inherent water-repellency of the treated garments.

C. O. C.

Extracting Cleaning Solvent from Dry-cleaned Articles (I p. 260).

#### VIII- DYEING

Solubilisation of Orange OT and Dimethylaminoazobenzene, M. W. Rigg and F. W. J. Lau. J. Amer. Oil Chem. Soc., 30, 14-17 (Jan. 1953).

Solubilisation data have been obtained for the amount of the two dyes Orange OT\* and dimethylaminoazobenzene (D.M.A.B.) in aqueous solutions of various wetting agents at 30°c. The results indicate that, using the technique described (in which the method of filtration is important). fairly reproducible results may be obtained, and that no dye is dissolved below the critical micelle concentration. The results are compared with those of other workers. Potassium and sodium laurates were found to produce the same solubilisation of either Orange OT or D.M.A.B., indicating that the solubilising species in solution depends on the nature of the long-chain ion. • o-Toluidine  $\rightarrow \beta$ -naphthol

Decrease in Fastness to Light of Dyeings due to Textile Processing. F. Weber. Textil Praxis, 8, 242-245 (March 1953).

A review of the factors affecting the fastness to light of various types of coloured textiles including certain combinations of both direct cotton and vat dyes on cotton, insufficient soaping of azoic dyes on cotton, wet fading of delustred cellulosic rayon, effect of certain crease-resist processes on cotton, aftertreatment of dyed cotton with cationic compounds, effect of prolonged boiling on certain premetallised dyes on wool, anomalous fading with certain yellow wool dyes on nylon, and effect of the cuprous ion technique in the dyeing of Orlon.

Chemistry of Diammonium Phosphate. W. B. Coleman. Amer. Dyestuff Rep., P 167-P 168 (16 March 1953).

Aq. soln. of diammonium phosphate have pH 7.9 and are suitable for conditioning wool before dyeing with acid The pH of an acid dyebath set with diammonium phosphate falls from 7.8 to ca. 5.8 during 2 hr. at the A. J.

Diammonium Phosphate in Wool Dyeing. J. N. Dalton. Amer. Dyestuff Rep., P 168-P 171 (16 March 1953).

Use of diammonium phosphate in an acid dyebath produces an initial pH of 7.8, falling to 5.8 during dyeing, thus enabling level dyeings and thorough penetration to

be obtained with aggregating acid dyes.

Dycing Wool with Metal-complex Dycs. T. Egger and R. Casty. Teintex, 18, 227-237 (April 1953).

A review of the theory and practice of dycing with Neolan and Cibalan (Ciba) dycs. In applying Neolans, unless Neolan Salt P is used or pale shades are dyed, the optimum pH is near to 2. This pH is achieved by using amounts of H<sub>2</sub>SO<sub>4</sub> varying from 5% (on the wt. of wool) with a liquor ratio of 10:1 to 24% with a liquor ratio of 200:1. It is shown by measurement of alkali solubility 200: I. It is shown by measurement of alkali solubility that the wool is affected equally under these extreme or H. E. N. under intermediate conditions.

Beam Dyeing—Controlling Beam Density by a Study of Flow Rates. J. H. Stradley. Amer. Dyestuff Rep., P 172-P 173 (16 March 1953). The rate of flow of dye liquor through a beam may be

determined from the amperage load on the pumping motor. One example is cited. A.J.

Tendering Action of Caledon Orange 2RT.

Whittaker. J. Textile Inst., 44, T 199 (April 1953). A reminder is given that Caledon Orange 2RT, referred to in a paper on degradation of dyed fabrics by sunlight (D. Ashton and M. E. Probert, ibid., T 1-T 11 (Jan. 1953)) is an unsafe dye. It is considered surprising that more tendering complaints were not received prior to 1932

Effects of Soaping Vat-dyed Material. J. Wegmann. Textil-Rund., 8, 4-9 (Jan.), 97-108 (March), 157-165 (April 1953).

Primarily, soaping develops the final shade and therewith the best fastness properties. Removal of loose pigment is only a secondary consideration. Complete development depends mainly on temp., time being of less importance. Addition of dispersing agents makes the process speedier and more effective, but does not permit the temp, to be lowered, e.g. to 80 c. In some cases it is important not to dry between oxidation and soaping.

Tables give the degree of change of shade on soaping and the effect of ironing and water-spotting on unsoaped and soaped dyeings. The changes which occur during soaping cannot be attributed to increased aggregation. facts support this view-(a) the dichroic behaviour of dyes in the crystalline state does not run parallel to their behaviour on the fibre; (b) dyes may exhibit changes in colour when in solution analogous to those which occur on soaping; and (c) reduction of soaped dyeings to the semiquinonoid state followed by reoxidation can reproduce the soaped form. It is suggested that the changes which occur on soaping are of a molecular nature, depending on variations in the degree of polarisation of the molecule. Vat dyes are divided into two classes depending on their behaviour on soaping-(i) those which deepen in colour because of increased polarisation and (ii) those which undergo a hypsochromic change because of decreased polarisation. The discussion ranges over adsorption colours on alumina; polymorphic modifications; fastness to heat, water-spotting, and other agencies; variation of colour with substrate; and analogous effects with azoic and sol. azo dyes. It is shown that colour changes normally produced only by prolonged soaping can be obtained in a short time by immersion of e.g. an unsoaped dyeing of Cellophane with Indanthren Brilliant Scarlet RK in an aq. alcoholic soln. or emulsion of ethylene chloride, must be present, but other solvents may be used. Solvents of zero dipole moment are inactive. In some cases, dipolar substances give more complete development than soap; in others (e.g. Ciba Blue 2G or Cibanone Blue GF), the colour change is in the opposite direction to that obtained with soap.

Stripping Colour from Dyed Hydrophobic Fibres. A.T.C.C. Philadelphia Section. Amer. Dyestuff Rep., 42, P 190-P 205 (30 March 1953).

Description of a series of experiments to ascertain the ease of colour removal from nylon, acetate rayon, Orlon, Dacron, dynel, Saran, etc. Stripping procedures are described, and the assessment is based on (1) amount of colour removed, (2) effect of stripping on redyeing, (3) effect on tensile strength, (4) effect on light fastness. The following stripping recommendations are made: ty Orlon—10% NaClO<sub>2</sub> adjusted to pH 3-4 with HNO<sub>3</sub>; 81 Orlon—acidified 5% NaClO<sub>2</sub>; Dacron—two-bath treatment, acid formaldehyde-sulphoxylate followed by NaOCl or NaClO<sub>2</sub>; dynel—NaClO<sub>2</sub> plus HNO<sub>3</sub> or quater-nary ammonium salt; Saran—NaClO<sub>2</sub> in presence of HNO<sub>3</sub> or benzoic acid; Acrilan—neutral NaOCl; acetate rayon-large volume of soap soln.; nylon-two-bath process, Zn formaldehyde-sulphoxylate followed NaClO<sub>2</sub>; X 51 fibre—NaClO<sub>2</sub>. A. I A. H.

Evaluation of Carriers for use in the Application of Acid Dyes to Dynel. J. H. Cox. Georgia Institute of Technology: Amer. Dyestuff Rep., 42, 134 (2 March 1953).

The quant, effect of assistants which swell and/or care dyes to dynel (and similar synthetic fibres) has been studied from the viewpoints of - (i) dyebath exhaustion, (ii) skein shrinkage, (iii) breaking strength, and (iv) light fastness of the dye. The experimental procedure is outlined. The best dyebath exhaustion was obtained with \$\beta\$-naphthol, p-phenylphenol,  $\beta$ -naphthylamine, and p-chlorophenol. Except in the case of p-chlorophenol, concn. > 3% led to shrinkage and loss of yarn breaking strength, whilst  $\beta$ -naphthylamine renders the dye fugitive to light. Resorcinol, quinol, Naphtol AS, and p-phenylenediamine are of doubtful value as carriers. In conen.  $\gg 4\%$ sulphuric, formic, salicylic, and benzoic acids give efficient dyebath exhaustion, and do not adversely affect yarn shrinkage and breaking strength, or light fastness of the J. W. D.

PATENTS

Improving the Affinity of Acrylonitrile Polymers for Monsanto.

Fibres of acrylonitrile polymers or copolymers have their dyeing properties greatly improved on treatment with NN-dimethylacetamide or NN-dimethylformamide. The dry fibre may be treated with either of the above agents, washed, and then dyed, or the dye may be dissolved in the treating agent; alternatively, the wet fibre may be treated with an aqueous solution of the agent. C. O. C.

Aqueous Pigmented Dyeing Composition. Imperial

Paper & Color Corpn. USP 2,614,086 Good fastness to rubbing and dry cleaning is obtained by using as the binder a condensate of melamine (1 mol.), H-CHO ( < 7.5), glycerol (1-4), and a hydroxyalkylamine (1-3) made by heating the constituents together until a clear homogeneous soln, is obtained, bringing to pH 0.5 2.0 with mineral acid, and then bringing to pH 6.7 before the resin precipitates or gels.

Multicoloured Aluminium (IX this page). Organic Anion Binding by Denatured Bovine Serum Albumin (XII p. 270).

#### IX-PRINTING

Studies in Textile Printing. IV—Some Factors involved in Vat Printing. R. D. Greene and F. Fordemwalt. Amer. Dyestuff Rep., 42, P 141 P 146 (2 March 1953).

Vat dye printing is discussed in terms of the individual steps in the process of application. The evaluation of prints by a method previously published, to which references are given, shows that the colour strength of a print alone does not necessarily indicate the true monetary value of the printing process involved. A method is described whereby the effects of the viscosity, thixotropy, and wetting characteristics of a printing paste are encompassed in a single measurement; the method is based on a measurement of the time required for the paste to penetrate a rather dense fabric under various pressures. The apparatus is illustrated. Surface tension effects, preventing penetration, become important under certain conditions, such as those of low printing pressure and compact fabric construction. Sedimentation, dyeing, and evaluation trials indicate that there is an optimum range of particle sizes for vat dyes, in terms of colour develop-ment. Experiments with Vat Pink FF show that fine, elongated particles of dye lead to strong, solid prints and high printing efficiency. The print loses strength and eventually becomes skittery as the dye particles become more massive, as may occur in a paste during storage. The particular type of crystal growth which occurs under such conditions determines whether or not the print is satisfactory. There are few data relating to the effect of drying prior to ageing, and there is scope for profitable investigation. A method is described, and the apparatus illustrated, whereby the rate of absorption of moisture by different printing gums from an atmosphere of steam has been determined. Alkaline gum pastes absorb moisture readily and do not appear to require additional deliques cents, though such agents may bring benefit through other causes such as solubilising action on leuco vat dyes. During ageing the leuco form of the dye must diffuse through the gum in order to reach the fibre surface, and a method for determining this rate of diffusion is described and illustrated. The rate of diffusion depends less on the dye than on the composition of the gum. Microscopic and photo-micrographic studies of printing pastes and dyed fibres can reveal the causes of poor fastness to rubbing, and the discussion of a typical case is suitably illustrated by photomicrographs. J. W. D.

du Pont Vat-print Development Process. Saville. Amer. Dyestuff Rep., 42, P 272 P 274, P 278 (27 April 1953).

A patented process for printing and developing vat dye prints consists in printing unreduced vat dye and drying, then padding the cloth with thickened NaOH-Na, S,O, using an engraved pin pad roller, and finally passing immediately into a steamer. The success of the process depends on having the entrance to the steamer in close proximity to the padding machine, so that the cloth enters the steam within 3 or 4 sec. of the application of reducing agent and alkali. The recommended steamer takes the form of an inverted U-tube with the exit leg immersed in the first tank of an open soaper, which makes an effective water seal. The cloth during its passage through the steamer is supported on the reverse side to prevent marking off. Alkali padding, steaming, oxidation, and soaping are carried out as a continuous process. The paper gives a preliminary short survey of existing vat printing techniques.

Progress in the Manufacture of Printing Screens. P. Welcker. Teintex, 18, 161-167 (March 1953). A review of various processes employed in the manu-

facture of screens, including several commercial recipes.

Photographic Reproduction in the Screen Printing of Textiles. A. Franken. Textil-Rund., 8, 113-118 (March 1953).

The possibilities and limitations of the process and a description of the apparatus used. H. E. N.

#### PATENTS

#### Binders for Printing with Pigments. C. Bener.

BP 691,541 Prints having good handle and exceptional stability to wear and aftertreatments are obtained with pigments using as the binder an aqueous dispersion of a vinyl butyral resin which is inert to water in absence of a curable synthetic resin.

Printing on Leather and other Anisotropic Materials. R. M. Colclough-Hoey,

In the "blocking" of leather, the pigment is bound to the leather or other surface to be printed by a flexible acrylic or vinyl resin, present preferably as a layer between the leather and the pigment. The process is suitable for leather which has subsequently to be distorted, e.g. to form shoe uppers, as the printed design will not crack, nor will it flake off in use.

#### Multicoloured Aluminium. Sinclair & Valentine Co. USP 2,614,912

The aluminium is printed in one or more colours, if more than one colour is used then portions of the designs overlap, is then dyed in a bath which contains no solvent for the printed designs, the surface is sealed by treatment with boiling water, and finally the residual ink vehicles are dissolved away. If desired a colourless resist may be C. O. C. applied before dyeing.

Colour Filters for Use in Two-colour Additive Systems of Colour Photography. Olympia Films BP 690,702 (London).

A mixture of C.I. 665, 748, and 749 is used for dyeing gelatin for filters having red-orange spectral transmission,

A mixture of C.I. 640 and 714 is used for filters having greenish-blue spectral transmission.

A mixture of C.I. 89, 150, 151, and 749 is used for orange red filters.

A mixture of C.I. 714, 925, and 1122 is used for yellowish-

green filters.

The chromaticity co-ordinates for taking and projecting e "red" and "green" colour selection records are the "red" and specified. C. O. C.

#### X-SIZING AND FINISHING

Effect of Conditioning on Crease-recovery Angle. A.A.T.C.C. Piedmont Section. Amer. Dyestuff Rep., 42, P 268-P 271 (27 April 1953).

Resin-treated and untreated fabrics are shown to give improved crease-recovery angles after extended conditioning, and if allowed to condition sufficiently long untreated fabric gives crease recovery equal to treated fabric. However, the lengthy conditioning period (several weeks) for untreated fabrics is impracticable, and herein lies the advantage of a resin treatment. Even so, the usual 24-hr. conditioning period is considered too short for optimum crease recovery, and a treatment comprising three rapid and consecutive dry cleanings before the 24-hr. conditioning period is advocated.

Hydrolysis of Methylenediurea. J. I. de Jong and de Jonge. Rec. Trav. chim., 72, 202-206 (March 1953).

The hydrolysis of methylenediurea to urea and formaldehyde is unimolecular. The rate of reaction is proportional to [H+] from pH 3 to pH 5, and the activation energy is 19-5 kg.cal./mole.

Kinetics of the Reaction between Hydroxymethylurea and Methylenediurea. Idem. Ibid., 207-212.

The condensation reaction between hydroxymethylurea and methylenediurea is bimolecular. The rate constants are directly proportional to [H<sup>+</sup>], and the activation energy is 15 kg.cal./mole.

Reaction of Methylenediurea with Formaldehyde. Idem. *Ibid.*, 213-217.

The condensation reaction between methylenediurea and formaldehyde is bimolecular and the rate is proportional to [OH-], but the reaction may also be catalysed by acids. The activation energy is 15 kg.cal.mole, and the rate constants have the same values as those for the reaction between urea and formaldehyde (ibal., 71, 643 (1952)).

Silicones in the Textile Industry. C. E. Gibson and F. L. Dennett, Amer. Dyestuff Rep., 42, P 275-P 278 (27 April 1953).

Description of the uses of silicones in the textile industry, particularly with regard to the water-repellent properties they confer.

A. H.

Educational Order Trial on 10-5-ounce Shrink-resistant Wool Flannel Shirting. L. I. Weiner and F. J. Rizzo, Amer. Dyestuff Rep., 42, 125-134 (2 March 1953).

In 1943 anti-shrink processes were made mandatory for hosiery and other knitwear supplied to the American forces. Years of subsequent investigation culminated in the crystallisation of suitable test methods and a tentative specification for 10-5-ounce shirting, by the Quartermaster Corps. In order to obtain the fullest information both for the Quartermaster Corps and for the various manufacturers, an educational order programme was initiated for 40,000 yd. of treated shirting, in which no single manufacturer was awarded more than 2000 yd. The widest possible range of processing resources were included when the contracts were placed. Wet chlorination processes included—(1) Harriset, in which the active chlorine derives from hypochlorite; (2) Kelpic, in which chlorination is by a mixture of hypochlorite and free chlorine in H<sub>2</sub>SO<sub>4</sub> soln.; (3) Kroy, a rapid, wet halogenation; (4) Sanforlan, an alkaline batch chlorination using KMnO<sub>4</sub> as adjunct; and (5) Schollerize, in which a buffered soln, of calcium hypochlorite is used. Resin treatments included- (1) Durlana, which uses a soap emulsion of a prepolymerised resin; (2) Koloc, based on an aq. emulsion of a prepolymerised resin; (3) Lanaset, in which the material is padded through a colloidal soln, of resin; and (4) Resloom, in which the resin is padded on the dry fabric. Manufacturers were given guidance in connection with difficulties which might arise when the shrink-resisting treatment preceded weaving. Methods for carrying out tests for shrinkage, stiffness, ageing, alkali-solubility, colour, and colour-fastness were detailed, and are briefly described in the paper. The results are discussed at length, and the extent to which individual requirements were met by the manufacturers is indicated. It is concluded that the production of this material appears both practical and feasible for military procurement. Production experience subsequent to the completion of these orders confirms this impression. J. W. D.

Eulan FL. Dr. Drapal. Textil-Praxis, 8, 330–332 (April 1953).

Discussion of the properties and application to wool of Eulan FL (FBy). The affinity of Eulan FL for nylon, the effect of temp. on its affinity for wool, and its quantitative estimation on the fibre are also considered. B. K.

PATENTS

Setting Synthetic Fibres in a Molten-metal Bath. Lansil. BP 691,370

Fabric formed of nylon or other synthetic fibre is set by being passed through a bath of metal at > 160°c. Nylon is set by an immersion of 2 sec. at 235°c. C. O. C.

Dimensional Stabilisation of Regenerated Cellulose Fabrics. American Viscose Corpn. BP 690,875
The material is treated with an aqueous solution at pH 1·2–1·8 of a water-insoluble alkali-soluble cellulose ether and a substance which is converted by heat into a water-insoluble resin, a mineral acid, and a salt thereof, dried, and baked. There is no loss in tensile strength and

there is no discoloration. The solution may be made by dissolving the cellulose ether and the heat-convertible substance, e.g. glyoxal, in aqueous NaOH and then bringing the solution to the desired pH by adding a strong mineral acid, e.g. H<sub>2</sub>SO<sub>4</sub>.

C. O. C.

Durable Mechanical Effects on Fabrics. Joseph Bancroft & Sons Co. BP 690,237

Fabrics of cellulose or cellulose derivatives containing hydroxyl groups are treated with bishydroxymethylethyleneurea, bishydroxymethyl-1:2-propyleneurea, or like bishydroxymethyl derivatives, mechanically printed while still moist, e.g. glazed or embossed, and then heated so that the cellulose will react with the bishydroxymethyl derivative. The latter reacts with the cellulose without polymerisation. This allows much greater latitude in the amount of finishing agent that may be used and gives greater durability with lower loss in strength than when thermosetting resins are used.

C. O. C.

Aminoplast Flame-resisting Composition. American Cyanamid Co. BP 690,291

Cellulose and wool textiles are given both a permanent flame-resisting finish, and also crease and shrinkage resistance, by treating them with an aqueous mixture of (1) a heat-curable condensate of formaldehyde with melamine, urea, an alkylated hydroxymethylmeamine, or an alkylated hydroxymethylmea (2–20 parts by wt.), (2) hydroxymethyldicyandiamide (10–35), and (3) an oxygen-containing acid of phosphorus (equivalent to 5–30 parts of 85% orthophosphoric acid), C.O.C.

tenacity cellulose acetate or other organic ester of cellulose by winding it on to a perforated tube and then passing the saponifying agent through it. C. O. C.

Cold-waving Solutions. Gillette Safety Razor Co.

Addition of ammonium carbonate and/or bicarbonate to commerical aqueous solutions of thiols, e.g. ammonium thioglycollate, enables them to be used at pH 7-7-9-9. The rate of splitting of the disulphide bonds is increased, and there is less tendency to overtreat degraded bair.

Permanent Waving. Henkel & Cie. BP 690,866
An aqueous solution of a salt of a mercaptobutane-sulphonic acid is used to split the disulphide bonds.

Cloth Carbonising. J. Dungler.

The acid-impregnated cloth has jets of a hot gaseous medium, e.g. superheated steam, violently projected against it so as to cause instantaneous evaporation of the water in all parts of the cloth, carbonising being then effected by continuing to treat the cloth with the hot gaseous medium.

C. O. C.

Nylon Tyre-cord Fabrics. Wingfoot Corpn. BP 690,165
Nylon tyre-cord fabric is stabilised by impregnating with a solution of a thermosetting resin, partly drying, and then heating at just below its m.p. for a short time whilst the material is stretched to remove the potential elongation introduced during the coating process.

W. G. C.

Coated Nylon Fabrics. ICI. BP 690,706
A colourless conting having excellent resistance to solvents, acids, alkalis, and detergents is given by applying a solution containing (1) a polyester from adipic acid and ethylene glycol and glycerol, and (2) an aromatic hydrocarbon diisocyanate, and then heating. The resultant coating is free from cracking or crazing even when creased at very low temperatures, the creases being removed by simple hot ironing.

C. O. C.

Fabrics having Active Agents thereon. J. Lindenmeyer.

BP 689,565

The active agent, a.g. activated carbon or collaborations.

The active agent, e.g. activated carbon or colloidal metal, is incorporated into the carrier while the latter is still in a plastic or mouldable state. This results in very even impregnation of the fabric with the agent. C. O. C.

Decrease in Fastness to Light of Dyeings due to Textile Processing (VIII p. 267).

#### XI-PAPER AND OTHER CELLULOSIC **PRODUCTS**

Action of Cold Concentrated Sulphuric Acid Solu-

tions on Carboxymethyl Celluloses. T. E. Timell. Svensk Papperstidning, 56, 245-250 (15 April 1953). 3-Carboxymethyl glucose was synthesised, and treated with conc. aqueous acid solutions at room temp. As the product remained unchanged in all cases, it was concluded that no carboxymethyl groups were split off. Carboxymethyl cellulose treated similarly underwent rapid depolymerisation followed by a preferential removal of low-mol.wt. material containing a higher proportion of substituent groups than the remaining portion. These results invalidate the conclusion previously drawn that carboxymethyl cellulose could be de-etherified by cold cone, sulphuric acid solutions. S. V. S. cone, sulphuric acid solutions.

Mixed Ethyl Cellulose-Butyl Hydroxyethyl Cellulose Films. H. C. Haas, L. C. Farney, and C. Ind. Eng. Chem., 45, 564-566 (March 1953).

Although most high polymers are mutually incompatible, it has been found that butyl hydroxyethyl cellulose containing 1.5 ethylene oxide and 2.0-2.1 butyl groups per anhydroglucose unit is compatible with ethyl cellulose in all proportions. Perfectly clear mixed films are obtained by casting from a common solvent, and film properties are linear functions of film composition. As the two materials differ appreciably, a wide range of mechanical properties may be obtained on blending. The butyl hydroxyethyl cellulose may be used as polymeric plasticiser for ethyl cellulose. W. K. R.

PATENTS

#### Bleaching Non-homogeneous Cellulosic Waste Materials. A. B. I. Azienda Brevetti Industriali. BP 691,564

Non-homogeneous fibrous pulp containing ordinary cellulosic fibres and particles of mechanical pulp, with rough non-cellulosic elements, is mechanically homogenised and simultaneously washed, without previous delignification, and then treated with chlorine in two stages-(i) excess of chlorine introduced into the mass at pH < 3, and (ii) alkali added to neutralise completely the residual chlorine. The first stage may be limited to 2 hr. duration, and a second chlorine treatment may be given, with reduced quantities of chemicals.

8. V. S.

#### Sizing and Bleaching Paper on the Papermaking Machine. Buffalo Electro-Chemical Co.

USP 2.613.579 Alkaline peroxide soln, and an inert wax size are added simultaneously to the formed sheet as it passes through the machine at a point where it still contains water. This makes it possible to bleach the paper and simultaneously to confer on it a controllable degree of water resistance.

#### Treatment of Papermaking Fibres with Organosilicon Compounds. Cowles Chemical Co BP 689,604

To improve the wet strength and water resistance of paper, the fibres are impregnated with an aqueous alkaline solution of the products of the alkaline hydrolysis of an organotrihalogenosilane, the pH of the solution being reduced to  $\gg$  11-0 (10-5–11-0) before or after it is applied to the fibres to precipitate an insoluble organosilicon compound on the fibres, and the impregnated fibres being cured at an elevated temperature, e.g. 95-120°c., to dry the sheet and complete the polymerisation of the com-pound. S. V. S.

Fireproofing Paper. T. W. Rannila. BP 690,434 Fireproof fibrous sheets from combustible fibrous material are manufactured by applying a mixture of a non-volatile, non-combustible inorganic ebloride, e.g. MgCl<sub>2</sub>, an inert powder, e.g. magnesite, and an organic adhesive which is insoluble or poorly soluble in water by spraying a suspension or solution on to the wet stock on the wire of a paper machine. Ammonium chloride, ammonium phosphate, or sodium carbonate may be added singly or together to improve the fire-resistance of insulating board.

#### XII-LEATHER: FURS: OTHER PROTEIN MATERIALS

Amide Nitrogen of Collagen and Hide Powder. J. M. Cassel and E. McKenna. J. Amer. Leather Chem. Assocn., 48, 142-154 (March 1953).

Amide nitrogen of collagen, hide powder, and gelatin, determined by estimating the NH<sub>3</sub> liberated by acid hydrolysis over an extended period and extrapolating back to zero time, is shown to be 6.0, 3.8, and 1.9 mg./g. protein respectively. Amide hydrolysis in collagen by 0.2 N-HCl at 100°c is of the first order until 90% of the reaction is complete; the activation energy is 18-3 kg.cal. per mole. Amide N is liberated more rapidly from collagen than from egg albumin; the catalytic effect of the Na salts of some mono- and di-basic sulphonic and sulphuric acids, e.g. sodium lauryl sulphate, on this acid hydrolysis is greater with wool and egg albumin than with collagen. Amide N in collagen is mainly asparagine rather than glutamine J. W. B.

Decarboxylation of Collagen. R. L. Sykes. Nature, 171, 654-655 (11 April 1953).

Lithium aluminium hydride effects a more extensive reduction in -COOH group content of collagen than does treatment of methylated collagen with methylmagnesium iodide. Some reduction of the amide groups is also observed J. W. B.

#### Organic Anion Binding by Denatured Bovine Serum Albumin. L. L. Uzman. Nature, 171, 653-654 (11 April 1953).

A study of the binding of an organic anion (methyl orange) by bovine albumin denatured with urea and guanidine hydrochloride shows that there is no decrease in the amount of anion bound to the albumin; rather denaturation causes an increase, depending on the statistical distri-bution of uncoiled, partly coiled, and "native" molecules in the denatured solution. The loss of binding affinity for organic anions observed with heat-denatured albumin probably represents a special case. J. W. B.

#### XIII—RUBBER: RESINS: PLASTICS

Factors affecting the Solubility of Polymers. P. A.

Small. J. Appl. Chem., 3, 71-80 (Feb. 1953).
Solubility of a polymer in a non-polymeric liquid depends mainly on the heat of mixing. Where no polar forces operate, the cohesive energy densities (C.E.D.) of polymer and solvent must be close; a method is given for estimating the C.E.D. of polymers from a set of additive constants which correspond to various structural features of the molecules. Values of solubility parameters calc. in this way agree well with those obtained by swelling measurements. The solubility of polyvinyl chloride in several solvents is considered, and correlated with the C.E.D. of the solvent and its hydrogen-bonding ability. The discussion is on a thermodynamic basis, and factors The discussion is on a thermosymmus such as molecular size, shape, and flexibility are not considered.

J. W. D.

Polyelectrolytes. A. Katchaksly. Endeavour, 12, 90-94 (April 1953).

A review of some properties of synthetic polyelectrolytes such as polyacrylic acids and polyamines. Osmotic properties of solutions depend on the degree of dissociation of the polyelectrolyte. Strong electrostatic fields around the polymer molecules strongly affect oppositely charged ions, restricting their movement and diminishing their contribution to osmotic properties. Osmotic pressures of polyelectrolyte solutions and activities of counter-ions decrease with increasing degree of ionisation of polyelectrolyte. Interaction between two oppositely charged polyelectrolytes may lead to mutual precipitation. Specific viscosities of polyelectrolyte solutions also vary with degree of ionisation. At low degrees the chains are tightly coiled. At degrees of ionisation of 10-20% the chains behave as randomly kinked coils. At higher degrees full extension of chains occurs. Synthetic cross-linked polyelectrolyte gels show expansion and contraction on addition of alkali and acid respectively because of changes in ionisation and resulting extension of chains. W. R. M.

Polymerisation of Vinyl Monomers in Aqueous Solution by Diazonium Salts. W. Cooper. and Ind., 407-408 (25 April 1953).

When diazonium compounds are decomposed by metallic ions, the polymerisation of vinyl monomers such

as methyl acrylate or acrylonitrile is imitiated after an induction period which is inversely proportional to  $[RN_2X]$  and  $[Metal\ ion]$ , and proceeds rapadiv. Tables are given indicating that the rate of polymerisation are given indicating that  $\alpha \in [\mathbb{R}^n]$ ,  $[\mathbb{R}^n]$ ,  $[\mathbb{R}^n]$ , where M is the monomer (acrylonitrile in the example quoted). This rate of polymerisation, though of the expected order for the diazonium compound, is higher than expected for Fe2+ and monomer concn., and this is attributed to heterogeneous polymerisation leading to the shielding of growing polymer chains by occlusion in insol. polymer. Studies with both Fe and Cu compounds show that radical formation is not initiated by the metal ion in its higher valency state. The polymerisation reaction has a low energy of activation (ca. 10 kg.cal./mole), and that of the initiation step is even lower (ca. 6-10 kg. cal./mole). Slight non-reductive decomp, also occurs, and whilst the formation of free radicals is a major reaction, there is as yet insufficient experimental evidence to prove that it is the only one. At high (5 s.) acid conen, the yield of polymer is greatly decreased, and it appears that non-radical decomp. predominates.

J. W. D.

#### PATENT

Finishing Sheets of Thermoplastic Material. E. C. Garcia and J. C. Garcia. BP 690,285

The material is pressed in direct contact with hot rigid engraving plates, preferably made of glass, the pressure being applied to the plates through a flexible cushioning material softer "in thermic function" than the material being pressed. According to the type of engraving plate used, the finish obtained may be brilliant, semi-brilliant, dull, or composite, or may be embossed. C. O. C.

Producing Wrinkle Texture on Non-wrinkling Films

(V p. 264). Mixed Ethyl Cellulose-Butyl Hydroxyethyl Cellulose Films (XI p. 270),

#### XIV-ANALYSIS; TESTING; APPARATUS

Symposium on Modern Methods of Analytical Chemistry. Anal. Chem., 25, 356–358 (Feb. 1953). Abstracts of 13 papers read at the Sixth Annual

Abstracts of 13 papers read at the Sixth Annual Symposium on Modern Methods of Analytical Chemistry at Louisiana State University on 2nd-5th Feb. 1953. Each deals with a generally applicable technique. J. W. D.

Temperature-indicating Paints, D. M. Kiefer. Ind. Eng. Chem., 45, 13A, 15A (March 1953).
A range of 28 coloured materials is available which

A range of 28 coloured materials is available which change colour at definite temperatures between 104°F, and 1832°F, with an indicating accuracy of ± 9°F. The Thermocolors (Basf) include materials which on being heated liberate H<sub>2</sub>O, NH<sub>3</sub>, and CO<sub>2</sub>, salts of Cu, Ni, Co, Cr, and Mo, ammine salts, etc. Some pass through as many as four distinct changes at progressively higher temperatures. Most are affected by H<sub>2</sub>S, and a few by NH<sub>3</sub> and SO<sub>2</sub>. The colour change is influenced also by the length of time at high temperature. The coloured materials are applied by mixing with alcohol and spreading the resulting paint thinly on the test surface.

W. K. B.

Colorimetric Microdetermination of Nitrogen. R. A. Boissonnas and C. H. Haselbach. Helv. Chim. Acta, 36, 576-581 (April 1953).

A new micromethod for the simultaneous determination of Kjeldahl nitrogen in numerous fractions obtained e.g. by chromatography or countercurrent distribution of proteins. Samples containing 1–20 µg. N are digested with 0-15 c.c. H<sub>2</sub>SO<sub>2</sub>, and neutralised with LiOH and Li acctate in 20% methoxyethanol, giving pH 4-6. Ninhydrinhydrindantin reagent is added, and on heating forms a blue cpd. with NH<sub>3</sub>. This is estimated colorimetrically. H. E. N.

Microgram and Submicrogram Determination of Phosphate—Applications of Sealed-tube Digestion and Capillary-cell Spectrophotometry. F. L. Schaffer, J. Fong, and P. L. Kirk. Anal. Chem., 25, 343-346 (Feb. 1953).

The many precautions and refinements essential in carrying out the acid digestion of samples of micro- and submicro-gram size are described and discussed. The molybdenum blue complex is extracted by means of octanol or a 1:1 mixture of isobutanol and benzene, and the colour estimated spectrophotometrically at 725 m $\mu$ ., capillary absorption cells being used for the lower ranges. Procedures are described fully for the analysis of phosphorus in quantities ranging from a few down to 0-002  $\mu$ g.

J. W. D.

Determination of Sulphate and Sulphonate Anionactive Detergents in Sewage. P. N. Degens, H. C. Evans, J. D. Kommer, and P. A. Winsor. J. Appl. Chem., 3, 54-61 (Feb. 1953).

A modification of the method of Jones (J. Assoca. Off. Agric. Chem., 28, 398 (1945)), which is based on complex formation with methylene blue, may be used to determine very small amounts of anionic synthetic detergents rapidly. Though the effects of various interfering substances can usually be eliminated by a suitable choice of the vol. ratio of aq. and org. phases, and by the number of extractions, the effect of thiocyanate at conca. > 5 p.p.m. cannot be wholly eliminated in this way; two alternative methods of removal by chemical reaction are described. Sulphate and sulphonate detergents may be separately estimated by hydrolysing the sulphates in boiling acid soln. and measuring the detergent content before and after treatment. The procedures are given in full.

Volumetric Determination of Persulphate in the presence of Organic Substances, 1. M. Kolthoff and E. M. Carr. Anal. Chem., 25, 298-301 (Feb. 1953).

The iodimetric and ferrometric methods for the determination of persulphate have been critically studied and compaced, with particular regard to the possibility of interference from organic substances which may be present. The iodimetric method is preferred except in cases where the organic substance reduces iodine or oxidises iodide; good results obtain at all pH values from 7 downwards. The stoichiometry of the ferrometric method is affected by organic substances; the induced reactions causing the disturbances may be climinated by adding bromide to the reaction mixture. The recommended methods are given in detail.

J. W. D.

Ultraviolet Spectrophotometric Determination of Nitrites with p-Aminobenzenesulphonic Acid. J. M. Pappenhagen and M. G. Mellon. Anal. Chem., 25, 341-343 (Feb. 1953).

25, 341–343 (Peb. 1953).
The ultraviolet absorbance at 270 mμ. of the reagent before and after diazotisation by the nitrite present is measured. The method is much less sensitive than the Griess method (the two have molar absorptivities of 15,500 and 41,000 respectively), but it is applicable at rather higher conen., and is much more rapid in operation. There should be the minimum of organic matter present. An account is given of the effect of various factors such as conen., pH, diazotisation time, and the possible interference of other ions. The method is specified. J. W. D.

Determination of Phenolic Hydroxyl Values (Semimicro Method). H. Brunner and H. R. Thomas, J. Appl. Chem., 3, 49-54 (Feb. 1953).

Most methods for the determination of hydroxyl values are based on acetylation followed by back-titration of unused acetic anhydride with standard alkali; though satisfactory for oils and alcohols, they yield low and variable results with substances containing phenolic hydroxyl groups. Those factors which affect the accuracy of the method, e.g. conditions of hydrolysis and the completion of the acetylation reaction, have been studied. Phenolic acetates have been found to hydrolyse fairly rapidly during the hydrolysis of anhydrides (which is normally carried out after the acetylation reaction), thus accounting for the observed anomalies. Though periods ranging from 2 to 6 hr. are commonly used for the acetylation, the authors find that reaction is virtually complete after 45 min. with substances ranging from glycerol to phenol. These studies, together with a recommended semi-micro method for use with phenol samples (20-80 mg.), are fully described. In determining phenol and phenolic resin intermediate acid values, Neutral Red should be used as indicator, and phenolphthalein is to be avoided; with dark-coloured soln. the end-point must be determined J. W. D. electrometrically.

#### Rapid Method of Identification of Textile Fibres in Blends. D. Sassi. Amer. Dyestuff Rep., 42, P 236-

P 240 (13 April 1953).

The method is designed to afford rapid and unambiguous identification of all the textile fibres now in use (both singly and in blends), involving a minimum of equipment and manipulation; it is based upon three preliminary "sorting" tests—(1) Selective staining of the fibres with a dve mixture comprising a yellow acetate rayon dye, a direct red, and a neutral-dyeing acid blue dye; suitable dyes are indicated. (2) Close visual examination of the dyed (or stained) yarns opened up under a hand magnifying glass or, preferably, a simple microscope at ca. 200 diameters. (3) Chemical tests, solvent tests (which are best carried out under the magnifier), and supplementary colouring tests. The recommended methods of carrying out the first two of these preliminary tests are detailed, and a full list of supplementary tests under heading (3) is given. A table is given summarising the results of the preliminary staining procedure, and there is also an alphabetically arranged table of fibres which affords crossreference to it. J. W. D.

Measurement of Flammability of Consumer Textiles. E. P. Johnstone. Amer. Dyestuff Rep., 42, P 96-P 98 (16 Feb. 1953).

Variation in the preparation of test specimens before testing can appreciably affect the result of the test and could produce significant differences between the results from different laboratories. Factors which may influence considerably the burning time are moisture content due to exposure to humid air of oven-dried sample before ignition and improper handling of napped or brushed specimens (flattening of the surface fibres). The following are considered to have little effect on the burning ratedrying time (if > 15 min. at 105 c.), temperature of oven-dried specimen at moment of ignition, and time of cooling in desiccator. Results of experiments are summarised by the aid of graphs.

#### Determination of Alkoxyl Groups. III Determination of Methoxyl in Carbohydrate-containing Substances, G. Gran. Svensk Papperstidning, 56, 179-180 (15 March 1953).

By the trimethylamine method previously described (ibid., 55, 255 (1952) ) good methoxyl values were obtained in analysing a well defined test substance. Pure simple sugars gave no methoxyl. Groundwood and chemical woodpulp gave methoxyl values varying with the lignin as determined from the amount of chlorine required for bleaching. Wood gave somewhat lower values by the trimethylamine method than by the Vieböck and 8. V. S. Schwappach method.

#### Determination of Alkoxyl Groups. IV-Absorption Svensk Papperstidning, 56. Solutions, G. Gran.

202 203 (31 March 1953).

It is shown experimentally that aqueous solutions may safely be used for absorbing the alkyl iodides formed in alkoxyl determination by the method of Vieböck et al. should be buffered to pH 3-4 and contain 0-5-1 M-KBr to keep the bromine in solution. Compounds, such as citric acid, that react with bromine should not be used as buffers.

#### **Estimation of Small Amounts of Carbon Disulphide** and Acetone by Ultraviolet Colorimetry, S. N. Andreev and B. I. Gindina. J. Appl. Chem. U.S.S.R., 26, 104-108 (Jan. 1953).

Acetone and CS<sub>2</sub> are estimated in CCl<sub>4</sub> soln, by ultraviolet absorptiometry with the aid of selective filters developed by the authors and described in the same issue (pp. 85-88). The method is rendered much more sensitive for acetone by prior conversion of the latter into iodoform. The lower limits for quantitative estimation are given as 0.3 p.p.m. of acetone and 4 p.p.m. of CS<sub>2</sub>.

#### Identification of Orlon and Differentiation of Nylon and Perlon. W. Reif. Melliand Textilber., 34, 219

Orlon is insol, in acctone and in formic acid, but in boiling Sesolvan NK (Basf) it gives a clear soln., which remains clear on cooling. Both nylon and Perlon are sol. in formic acid, but the former is insol, in boiling Sesolvan NK, whereas the latter gives a colloidal soln., which sets to a gel on cooling. All three fibres can thus be distin-guished. In boiling Sesolvan NK Terylene is sol., but the

soln, sets to a gel on cooling; PeCe breaks up into a brown mass; whilst acctate rayon is sol., the soln, becoming very viscous on cooling. Orion can also be distinguished from other synthetic fibres by dry distillation and testing of the vapours evolved with paper impregnated with Cu acetate and benzidine acetate, which is turned blue by HCN H. E. N.

#### Tactual and Instrumental Ranking of Softeners on Starched and Unstarched Cottons. C. R. Stock and A. Hvizdak, Text. Research J., 23, 186-196 (March 1953).

Various methods for measuring the softening of cotton fabrics are discussed. It is shown that a procedure involving yarn removal is capable of detecting small changes produced by different finishing treatments on one kind of

### Electrometric Method for the Determination of Relative Opacities of Water Pigment Finishes. J. S. Mudd and F. E. Downs. J. Soc. Leather Trades

Chem., 37, 67-69 (Feb. 1953).

The ability of a pigment finish to hide grain defects in leather is difficult to determine by existing methods. The construction and use of an apparatus for use in conjunction with a standard E.E.L. reflectance spectrophotometer is described. Light from a 6-v. bulb passes through an annular photocell, through a glass cell containing the finish in known dilution, and is then reflected from a standard surface to impinge upon the photocell, passing again, in so doing, through the cell containing the finish. The galvanometer unit is adjusted to give a reading of 100 units when a white standard surface is used for reflection; this latter is then replaced by a black surface, so that any light formerly reflected by the white surface is discounted, and the galvanometer reading gives a direct measure of the relative opacity of the finish. The choice of dilution is somewhat arbitrary, but practical trials show that, when a liquid-holding cell 2.5 mm. thick is used, a concn. of 0.5% is sufficient; above this value the curve of relative opacity versus conen. becomes asymptotic. Differences are revealed which are not apparent from determinations of specific gravity or viscosity. A tolerance of 2% variation in the preparation of successive batches of the same finish is recommended. Relative opacities of ten colour finishes are given. There is a diagram of the apparatus.

#### PATENTS

Colour Density Analyser. J. M. Hall. USP 2,608,125 Apparatus for measuring the colour and colour density surfaces and transparencies.

Colour-matching Apparatus. American Cyanamid Co. BP 688,894

Apparatus for matching the tristimulus values of dyeings with predetermined dyes. C. O. C. Tensile Strength Tester. R. R. La Torre and T. W.

USP 2,608,857 George. The mounting head of a tensile strength testing machine enables the test specimens to be mounted in a geometrical relation to the head so as to eliminate undesirable stress C. O. C. variables.

Co-operative Research - Exposure Testing on a Co-operative Basis (V p. 263).

Viscometric Studies of Cellulose in Cotton in relation to

Mechanical Processing (VI p. 264).

Degradation of Cellulose during Mechanical Processing.

III—Comparison of the Evaluation by Viscometric Methods (VI p. 264).

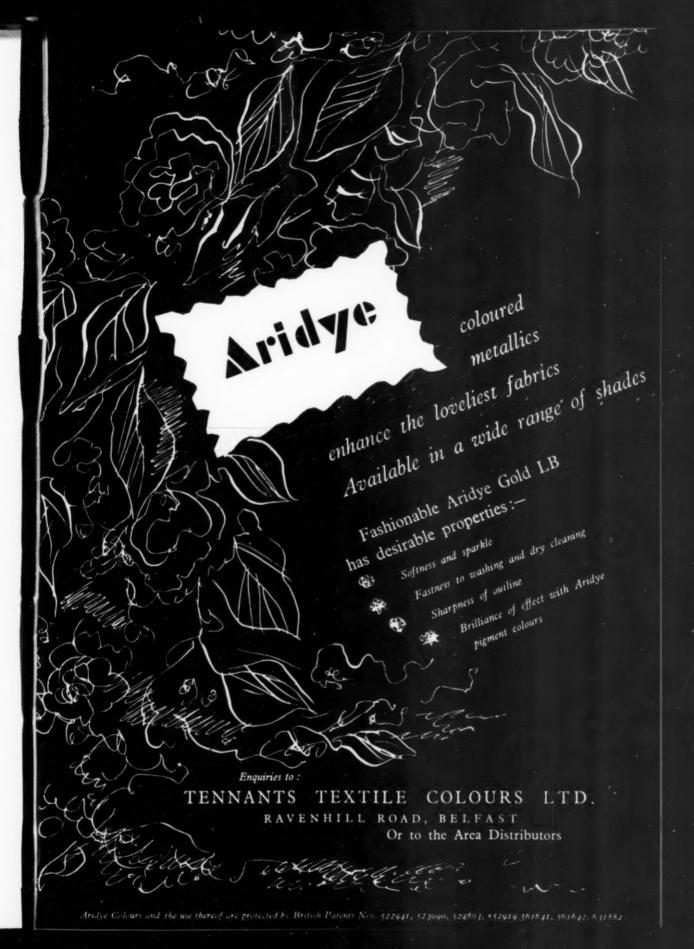
Separation of iso- and Tere-phthalic Acids by means of the Thallous Salts (VI p. 265).

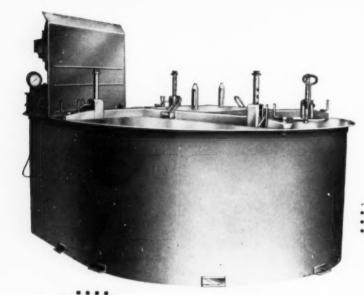
Eulan FL (X p. 269).

#### XV- MISCELLANEOUS

Control of Industrial Anthrax. E. Hailer and K. Heicken. Z. Hyg. Infektkr., 131, 219 (1950): Vet. Bull., 22, 673 (1952): J. Soc. Leather Trades Chem., 37, 100 (March 1953).

The sporicidal action of sodium sulphide is caused by caustic soda formed by hydrolysis. Sodium hydrosulphide and sulphuretted hydrogen are ineffective against anthrax spores. When mixed with calcium sulphide, the sporicidal action of sodium sulphide is diminished. C. J. W. H.







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1953 Tuesday 29th Sept.

Tuesday

27th Oct.

Dr. I. S. M. Walls (I.C.I.). The Dycing and Finishing of Fabrics containing the Newer Synthetic Fibres

Dr. G. H. Lister (Sandoz Products Ltd.). The Migration Properties of Acid and Chrome Dyestuffs 1953

Tuesday 17th Nov.

J. S. Ingham, Esq., M.Sc., F.R.I.C. (Marks and Spencer Ltd.). Problems of Colour Fastness and Finish

Tuesday Prof. P. D. Ritchie. Links between the 15th Dec. Textile and Plastics Industries

All Meetings in St. Enoch Hotel, Glasgow, at 7.0 p.m.

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Mr. J. Rogers, O.B.E., LL.D., chairman of the Company, in the course of his speech said—Your directors remain alive to the importance of adapting the company's operations and organisation to meet changing conditions. Perhaps I may be allowed to dwell a little longer on this aspect of our affairs.

aspect of our anairs.

Your company's activities range widely over the chemical field and indeed are not confined to that field, embracing as they do, for example, non-ferrous metals, "Lightning" fasteners, paints, leathercloth, and some semi-fabricated plastic materials. Its products are numbered in thousands and they are supplied to many industries. The organisational problems that confront your company are therefore much more complex than those experienced by undertakings concerned with the manufacture of one or two main products. Your company's research and development work is directed towards improving its existing manufacturing processes, developing new processes, developing new uses for its existing products, and to discovering new products and creating markets for them. On this work of research and development we are now spending at the rate of about £7,500,000 a year. Today we are making many products which were unknown before the war, and some of our methods of producing our older and more traditional products are very different and much more efficient than they were before the war. We can expect our new products to contribute an increasing proportion of the company's profits.

This process of change and development must go on if the company is to remain vigorous and prosperous, for the chemical industry still has great possibilities for expansion, and in expanding, it can contribute much to the national economy. Such expansion calls for more, not less, research and development and this work must be well inspired and well led. Not every research results in a new product or a better process, and it may take seven to ten years or even longer to pass from the first research result to commercial production.

Your company manufactures in many overseas countries, in most cases in association with nationals of the countries concerned; and, as our Annual Reports have clearly shown, our export trade is world wide. Its overseas interests are comparable in size and importance with the interests it has in the United Kingdom and are even more complex.

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The steadily improving efficiency of your company's operations means lower costs of production and these lower costs are reflected in the selling prices of our products. We have had to advance the selling prices of many of our products in recent years because of increasing costs of raw materials and rising wages and salaries, but we have been able to offset these cost increases to a considerable extent by savings resulting from improved methods and increased efficiency. As a result, our prices are, in general, lower than those ruling in America and in Europe.

You will remember that four years ago Lord McGowan told you that the whole Board of I.C.I. took the view that your company is not an appropriate subject for nationalisation. That remains the view of your board today, in whatever form state ownership is suggested.

Lord McGowan also said that the board would take all proper steps to oppose the nationalisa-tion of your company if the attempt were ever made. Let me renew that assurance. You will, I am sure, agree that it would be contrary not only to the interests of the stockholders and of those employed by your company but also to the interests of the country as a whole for I.C.I. to come under any form of state ownership.

"CHALLENGE TO BRITAIN"

I have just read the Labour Party's pamphlet called "Challenge to Britain". There is nothing in that document to cause me to change my view that any form of state ownership would be extremely

The main reason given in the pamphlet for this programme of public ownership is that chemical production must expand to keep pace with the enlarged requirements of other basic industries. Of course chemical production must expand, and this is exactly what has taken place in the industry as a result of the enterprise shown by those in it. My speech today emphasises the expansionist policy which I.C.I. has consistently pursued. I.C.I. is meeting every demand for chemical materials made upon it in the United Kingdom, and the average level of prices is lower than that in the U.S.A. and Europe. In addition its exports have increased very substantially in volume and in value since the war and are now running at over £1,000,000 per week. I can say without qualification that no company has a finer record of expansion and enterprise.

Why then should public ownership be suggested as a safeguard against possible shortages, which do not exist, and which will never occur, if private enterprise is permitted to go ahead? Surely the experience of the state-owned industries is convincing evidence that whatever else may be said about state ownership it is no guarantee that production will keep pace with increasing demand. I can only hope that those responsible for "Challenge to Britain" will give the whole subject further factual study and that in the long run wiser counsels will prevail.

Improvements in methods of producing our existing products and the reduction of costs go hand in hand with the development of new products. Your directors are also very conscious of the need to keep down the level of overhead expenditure. Special attention is being given to expenditure on central services, which is being kept to a minimum consistent with efficiency and progress.

#### CHAIRMAN'S RETIREMENT

I should like to finish on a personal note. This is the last occasion upon which I shall occupy this chair. As announced to the Press immediately after the Board Meeting on the 15th May, I am retiring both from the chair and from the board at the end of this month. I have been a director of this company both from the chair and from the board at the end of this month. I have been a director of this company since its formation 27 years ago and my association with Nobels, one of the constituent companies which were merged on the formation of I.C.I., goes back to the beginning of the century. May I take this opportunity of thanking my colleagues for all their help, their kindness and their co-operation during my long association with them and particularly for their support since I took over the chairman-ship from Lord McGowan two and a half years ago. You can be sure my successor. Dr. Fleck, will be given similar support. [The report and accounts were adopted.]



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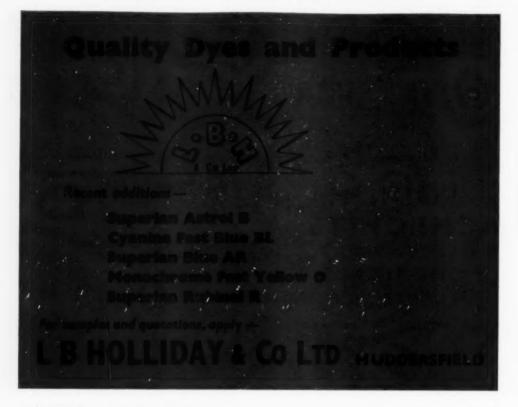
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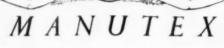


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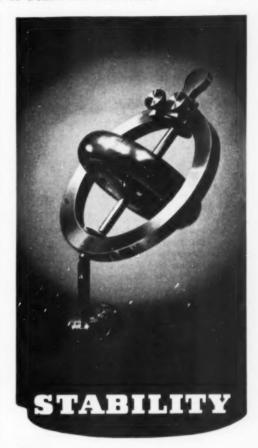
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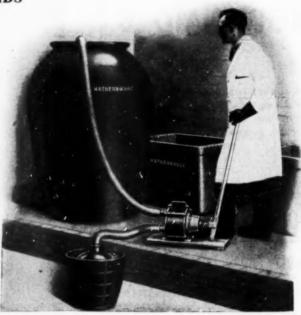
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